Faculty of Engineering and Science

On Potential of Natural and Artificial Particulate Mixtures as Reinforcements in Hybrid Bio-composites

Fong Ai Ling

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DECLARATION

To the best of my knowledge and belief this thesis contains no material previously published by any other person except where due acknowledgment has been made.

This thesis contains no material which has been accepted for the award of any other degree or diploma in any university.

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ABSTRACT

The interest in studying the biodegradable composites reinforced with natural fibers has been increasing exponentially. Therefore, efforts have been directed toward developing and characterizing natural fibers filled hybrid bio-composites where enhanced mechanical properties are being introduced to the materials through different combinations and modifications of constituent materials.

However, the challenge of the natural fibers and particulates reinforced composites is to eradicate the incompatibility between fibers and matrices, and the tendency to form aggregates during processing which causes poor interfacial adhesion. This further relates to poor mechanical properties with the composites. Hence, in this research, a hybrid composite material consisting a combination of rice husk ash particulates (RHA), sugarcane bagasse fibers (SCB), and nanosilica embedded into the epoxy matrix is developed. The SCB contains cellulose, which is the major framework component of its fiber structure. As the RHA particulates are in micron size while the nanosilica is nano-size, the nanosilica will be able to fill the smaller voids in the resin while the RHA particulates will fill bigger voids of the resin.

Thus, through this research, natural fibers and particulates filled hybrid bio-composite with enhanced mechanical properties (tensile and flexural properties) is–developed by hand-layup method. The interrelation behaviors between the matrix and the reinforcing phases will be obtained through characterizations and experimentation using tensile tests, flexural tests, scanning electron microscope (SEM), and XRD tests. These characterizations are carried out with various combinations of different weight percentages (1%-7.5%) of the reinforcing materials in order to establish the optimum reinforcing percentages for enhanced mechanical properties. For SCB short fiber composites, 1wt% of short fiber SCB composite exhibited better mechanical properties compared to other percentages while SCB particulate composite has better overall performance at higher weightage of SCB. Furthermore, for hybrid series composite, the improvement ranges from 11% to 63% for tensile strength and 18% to 37% for Young's modulus respectively compared to the pure SCB series composite.

It is envisaged that the results from this research will be useful for researchers in exploring the future potential applications of the hybrid composites in various fields such as the aerospace, automotive, and biomedical industries.

Keywords: Natural Fibers, Sugarcane bagasse fibers, Rice Husk Ash particulates, Epoxy Matrix, Hybrid Bio-composite Material, Nanosilica particulates.

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NOMENCLATURE

Nomenclatures	Description	Units
Pa	Pascal	kg/ms^2
wt	Weight	g
D	Density	g/ cm^3
Tg	Glass Transition Temperature	˚C
UTS	Ultimate Tensile Strength	N/mm^2

LIST OF ABBREVIATION

BRHA	-	Black Rice Husk Ash
RHA	-	Rice Husk Ash
WRHA	-	White Rice Husk Ash
SCB	-	Sugarcane Bagasse
HP	-	Hybrid Particulate
HF	-	Hybrid Fiber
SEM	-	Scanning Electron Microscope
XRD	-	X-ray powder diffraction
ASTM	-	American Society for Testing and Materials
C-SCRP	-	Chopped sugarcane/polyester composite
NAOH	-	Sodium Hydroxide
HDPE	-	High Density Polyethylene
SiO^2	-	Silicon Dioxide
SGF	-	Short Glass Fiber
PP	-	Polypropylene
SCF	-	Short Carbon Fiber
TEM	-	Transmission Electron Microscopy
USAXS	-	Ultra-small-angle X-ray Scattering
n-SiC	-	Nanosilicon Carbide
OH	-	Hydroxyl
TGA	-	Thermogravimetric Analysis
OMC	-	Organic Matrix Composites

PMC	-	Polymer Matrix Composites
MMC		Metal Matrix Composites
CMC		Ceramic Matrix Composites

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Fundamentals of composite

Composite is generally a type of structural material that are made up of two or more constituents which are combined together and are not soluble in each other (Al-Mosawi Ali, 2012). There are two groups of composite materials. The first group is categorized according to the matrix constituent. The main classes of these composites are Organic Matrix Composites (OMC), Metal Matrix Composites (MMC) and Ceramic Matrix Composites (CMC). Under Organic Matrix Composites, the classes of composites are further classified into Polymer Matrix Composites (PMC) and carbon matrix composite, which are also known as the carbon-carbon composites. While the second group is classified by the reinforcement forms, which consists mainly of fiber-reinforced composites, laminar/flakes composites and particulate composites (Kumar et al., 2015).

Polymer matrix composites are extensively utilized these days because they have high possibility of yielding a combination of high performance, good versatility, and processing advantages at a rational cost (Ornaghi et al., 2010). The commonly used polymers for composites applications are the thermosets. Examples of thermosets are epoxies, phenolic, polyurethanes, etc. While for thermoplastic, there are polypropylenes, polyethylene, elastomers and unsaturated polyesters matrices.

Generally, composites are also considered as cohesive structures that combine the matrix (primary phase) and the reinforcement medium (secondary phase).The matrix is utilized as a binder material to hold the fibers in position and also transmit external loads to the internal reinforcement (Chawla, 2012). Epoxies are one of the most common polymers and important matrices that are extensively used in fiber-reinforced composites. The families of polymer matrix are shown in Figure 1.1.

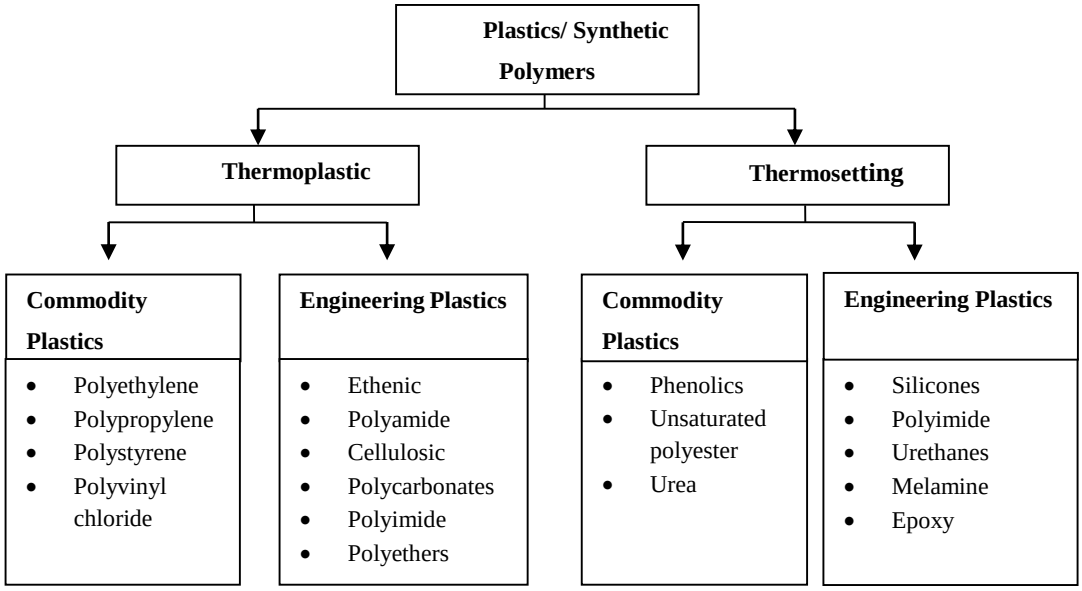


Figure 1.1 Families of polymers (Chawla, 2012)

The secondary phase, also known as reinforcing phase, can be found in the form of fibers, whiskers, particulates, or a variety of other geometries. While the matrix phase, which is the primary phase can be from any of the three fundamental material types which are polymers, metals, or ceramics (Callister, 2007). The former is typically possessing stronger, lighter and stiffer characteristic. Hence, the composite will possess better mechanical properties after reinforcement. The concept of composites is that the primary phase receives the load, and transfers it to the reinforcement material, which can withstand higher load. The significance here is that there are abundant matrix materials so as the reinforcement types. This reflects in the combination to be done in innumerable ways in order to fabricate a new composite with the anticipated properties (Sule, 2014).

Fibers are threads of reinforcing material and generally have circular cross-sectional shape. They are also found in tubular, rectangular and hexagonal shapes (Groover, 2002). Fibers are usually strong and stiff in terms of tension. This is due to the molecules of the fibers that are oriented in the longitudinal direction with small diameters; usually less than 0.01 mm (Kalpakjian & Schmid, 2010). They are desirable to be utilized as the reinforcing agent due to their filament form in majority of materials which is stronger than the primary form (Groover, 2002).

The composites that are reinforced with fibers are known as fiber-reinforced composites. Reinforcement is needed is to enhance the mechanical performance of

the composites. Fibers are preferable due to their lightweight characteristic as it reduces the weight of the overall product. Conventional fiber-reinforced composites are often produced with the reinforcement of carbon or glass fibers that are embedded into the matrices. These composites have good mechanical and thermal performance and, hence, are widely utilized in multiple areas.

Natural fibers, also known as bio-fibers, have been widely utilized as reinforcement in various composites. The underpinning reasons in the surge for applying natural fibers as reinforcements in composites are due to their renewability, biodegradability and low-cost in their applications (Cao et al., 2006). The utilizations and future potentials of these natural fibers which includes jute, straw, wood fibers, rice husks, sugar cane bagasse, bamboo cane, banana fiber, etc. have been studied (Taj et al., 2007). These fibers are typically composed of cellulose, hemicelluloses, lignin and pectin's, with minor extractives (Cerqueira et al., 2011).

Hybrid composites are composites that are having two or more types of reinforcing agents in the matrix (Sule, 2014). The combination of reinforcement is usually selected to produce a balanced composite in terms of strength and stiffness. It also provide dimensional stability, reduce cost and the weight of the material while enhance fatigue and fracture resistance (Sule, 2014). The hybrid fiber reinforced composite has been studied for various combination of reinforcing materials and matrices. Hybridization of composites that are reinforced with both natural fibers or synthetic fibers, are able to uplift the mechanical properties of the composite (Sule, 2014).

1.2 Layout of the report

This report contains 5 chapters which comprises of introduction, literature review, research methodology, results and discussions, and conclusions and future works.

- Chapter 1 defines the scope and structure of the thesis and the background of composite was also presented.

- Chapter 2 is the review on the different types of composites, both natural and synthetic are discussed. Moreover, the shortcomings and advantages of the natural fibers are also discussed. Thus, proposing that natural reinforcement with different shapes (ie: particulate and fibrous form) should be integrated into the composite matrix to produce a hybrid composite material.

- Chapter 3 discusses the research methodology for the fabrication of the composite, including the preparation and surface treatment of fibers are presented.

- Chapter 4 presents the results obtained are analyzed and discussed.

- Chapter 5 draws conclusion, feasible recommendation and future works that are related to the study are presented.

CHAPTER 2
LITERATURE REVIEW

2.1 Natural reinforcing materials

Natural fibers are defined as fibers which are not synthetic and characterized based on the types of sources in which these fibers are derived. Plant fibers are usually made of cellulose while animal fibers are made of proteins.

Contrary to the synthetic fibers in composite, natural fibers in composites are known in terms of the ability in offering environmental benefits such as lower pollutant and greenhouse gas emissions, improved energy recovery and end of life biodegradability of components (Tripathi. P 2015). Furthermore, the natural fiber reinforced polymer composites are reported to achieve U$2.1 billion in 2010 as its industry is being developed vigorously and estimated to grow at 10% worldwide for the next five years (Mohammed et al., 2015).

In general definition, natural fibers are naturally occurring fibers that are not synthetic, or in other words, man-made (Mohammed et al., 2015). It can be classified from various sources in which these fibers are derived. Douglas et al. (2014) reported that they can be sourced from three main original sources which are mineral sources, plant dry matters and animal fibers. Plant fibers are usually made of cellulose. Besides that, animal fibers are comprised of proteins. Chandramohan & Marimuthu (2011) have also reported the likelihood to extract natural fibers from mineral sources that are composed of naturally occurring fibers. The natural fibers are classified into six basic types, which are bast fibers, which are commonly found in jute, flax, hemp, ramie and kenaf. Furthermore, there are leaf fibers, which are found in abaca, sisal and pineapple and seed fibers, are found in coir, cotton and kapok. While kenaf, hemp and jute are from the category of core fibers while grass and reed fibers are found in wheat, corn and rice and all other types.

Three major structural parts of plant fibers are cellulose, lignin and hemicelluloses. Due to these three components of the fibers, the plant dry matters

are referred as lignocellulose (Douglas et al. 2014). The advantage of lignocellulose includes the safety features of these fibers which cause no threats to the surroundings (Satyanarayana et. al 2009). According to Jawaid et al.2011, lignocellulose fibers have less health damaging factors to the workers like skin irritation and lung cancer. Furthermore, they are also renewable materials and recyclable compared to the synthetic fibers which are non-renewable and costly. Cellulosic fibers such as sugarcane bagasse are easily obtained at minimal cost as they are considered as natural waste products.

According to Satyanarayana et.al (2009), glass fiber is found to be pricing at USD 1200-1800/tons while the plant fibers have an estimated price of USD 200-1000/tons. Furthermore, the fluctuations in the terms of the price of petroleum-based products are also reasons behind the increase of utilizations of the naturally occurring alternatives. It also aids in helping to increase the income of many developing countries, such as jute fibers which are found in abundance in Bangladesh and sisal fibers in Tanzania (Discover Natural Fibers 2009). Approximately 30 million tons of lignocellulosic fibers are produced and used in textile industry as well as packaging and composite materials, which used as parts in automobile, building materials, and sports equipment (Jamaid et al.2011). Moreover, for plants-based fibers also cause less abrasiveness to tool wears in terms of manufacturing aspects (Satyanarayana et al., 2009). The benefits and shortcomings of natural fibers are shown in Table 2.1.1.

Table 2.1.1: Advantages and disadvantages of natural fibers (Sreekumar, 2008)

Advantages	Disadvantages
1) Relatively lower specific weight	1) Poor resistance to moisture
2) Higher specific strength and stiffness compared to glass	2) Restricted maximum processing temperature
3) Thermal recycling is achievable	3) Variable quality, influenced by weather
4) Renewable resources and is biodegradable	4) Lower impact strength and durability
5) Lower cost in terms of fabrication/production	5) Poor fiber-matrix adhesion
6) Chemically friendly process methods, without wearing tools and free from skin irritation	6) Price fluctuation depending on the harvest seasons or agricultural politics
7) Good electrical resistance, thermal and acoustic insulating properties	

The natural fibers that are more commonly found and known in the world with the total world production are shown in Table 2.1.2 below. From the table below, it is shown that sugarcane bagasse is available in large amounts and it has shown that sugarcane bagasse is one of the main fiber source as it has the highest world production compared to others. Since sugarcane bagasse is so available, the focus is to use this available waste and turn it into resources.

Table 2.1.2: Natural fibers in the world and their total world production (Mohammed et al., 2015)

Fiber Source	**World production (10^3 ton)**
Sugar cane bagasse	75,000
Bamboo	30,000
Jute	2300
Kenaf	970
Flax	830
Grass	700
Sisal	375
Hemp	214
Coir	100
Ramie	100
Abaca	70

Table 2.1.3 shows the mechanical properties of commercially important lignocellulosic fibers that are potential fillers for reinforcement in composites. Mechanical properties that are stated in Table 2.1.3 are significant information for the fabrication of different composite. The mechanical properties of the product (composite), are significantly related to the mechanical properties of its reinforcement, the dispersion of fibers in matrix, and the efficiency of stress transfer from reinforcement to matrix.

Table 2.1.3: Mechanical properties of commercially important lignocellulosic fibers (Khalil et al. 2010)

Fibers	**D,Density (g/cm^3)**	**Tensile strength (MPa)**	**Young's modulus (GPa)**	**Elongation at break (%)**
OPEFB	0.7-1.55	248	3.2	2.5
Flax	1.4	800-1500	60-80	1.2-1.6
Hemp	1.48	550-900	70	1.6
Jute	1.46	400-800	10-30	1.8
Ramie	1.5	500	44	2
Coir	1.25	220	6	15-25
Sisal	1.33	600-700	38	2-3
Abaca	1.5	980	-	-
Cotton	1.51	400	12	3-10
Kenaf (bast)	1.2	295	-	2.7-6.9
Kenaf (core)	0.21	-	-	-
Bagasse	1.2	20-290	19.7-27.1	1.1
Henequen	1.4	430-580	-	3-4.7
Pineapple	1.5	170-1627	82	1-3
Banana	1.35	355	33.8	5.3

2.1.1 Sugarcane bagasse fiber

Sugarcane bagasse which is also known as *Saccarhum officinarum* is a plentiful agro-industrial waste that is utilized in various applications in countries such as Brazil (Tita et al.2002). While in South East Asia countries like Malaysia, sugarcane is one of the main crops in tropical region that covers approximately 34500 acres of plantation area (Salit 2014). The production of sugarcane is estimated at 1.3-1.6 million tons per year (Kadir & Maasom, 2013).

Bagasse is a waste product which is produced in boundless quantities by the sugar industries. Generally, one ton of sugarcane produces approximately 280 kg of bagasse, which is the fibrous residue that remain after sugar is being extracted from sugarcane (Sun et al. 2004). The breakdown of bagasse are 43.8% of cellulose, 28.6% of hemicellulose, 23.5% of lignin, 1.3% of ash and 2.8% of other components (Luz et al. 2007). Cellulose and hemicellulose are hydrophilic in nature while lignin is hydrophobic (Sun et al. 2004). The content of silica and carbon are approximately 9.78% and 90.22% respectively in sugarcane bagasse (Sun et al.2004).

By implementing the appropriate technologies, sugarcane bagasse can be treated as resource instead of waste as it can be processed into a set of supplies with national and international market potential, especially in composite materials (Contreras et al.2009). Hence, research activities are being carried out over the past decades in order to study the potential of the sugarcane bagasse fibers to be implemented in the composite. It has been considered as having a great approach in use as a reinforcing material in polymer composites.

Sugarcane bagasse fibers have relatively good in mechanical performance in terms of tensile strength, modulus of elasticity, density and elongation at break (Balaji et al., 2015). The mechanical properties of the fibers are affected by the age and source of the fibers. The different procedures or material used for surface treatment of these fibers also affects their properties. (S. Luz et al., 2007). Among all these factors, sugarcane fibers treatment shown to have great effect towards modifying the mechanical properties of the fibers (S. Luz et al., 2007). The studies about mechanical properties of untreated sugarcane bagasse fibers, modified and treated sugarcane bagasse fibers are presented in Table 2.2.1 and 2.2.2 respectively.

Table 2.1.1.1: Mechanical properties of untreated sugarcane bagasse fibers *(Vilay et al., 2008 Wirawan et al., 2010 Balaji et al. Satyanarayana et al., 2009 Trindade et al., 2005)*

Types of Modification to the SCB Fibers	**Elongation at Break (%)**	**D, Density (g/cm^3)**	**Tensile Strength (MPa)**	**Modulus of Elasticity (GPa)**	**References**
Untreated	4.03	-	96.24	6.42	(Vilay et al., 2008)
Untreated	-	-	170 – 290	15 – 19	(Wirawan et al., 2010)
Untreated	-	1.25	290	17	(Balaji et al.)
Untreated	-	1.12 – 1.15	180 – 290	15 – 19	(Satyanarayana et al., 2009)
Untreated	1.1	-	222	-	(Trindade et al., 2005)

Table 2.1.1.2: Mechanical properties of modified and treated sugarcane bagasse fibers (Vilay et al., 2008 Trindade et al., 2005)

Types of Modification to the SCB fibers	**Elongation at Break (%)**	**D, Density (g/cm3)**	**Tensile Strength (MPa)**	**Modulus of Elasticity (GPa)**	**References**
Sodium Hydroxide, NaOH treated	5.84	-	156.88	7.13	(Vilay et al., 2008)
Acrylic Acid, AA treated	5.59	-	229.01	8.09	(Vilay et al., 2008)
Oxidized Sugarcane Bagasse	0.7	-	126	-	(Trindade et al., 2005)
Oxidized Sugarcane Bagasse Reacted with Furfuryl Alcohol (FA)	0.9	-	238	-	(Trindade et al., 2005)

2.1.1.1 Sugarcane bagasse fiber- reinforced composite

Several studies have been carried out to understand the mechanical properties of the composite by embedding it with sugarcane bagasse fibers. Leite et al. (2004) have conducted a study to investigate the chemical and physical properties of sugarcane bagasse fibers by mixing it with phenolic resin. The phenolic resin was mixed with the cane pulp and the additives in a container. The mixture was homogenized and placed in a mold according to ASTM D638 standards in which shape and size vary according to assays. The results obtained with the tensile experiments demonstrate that strength increases with the proportion of fibers, reaching a maximum value at 29% fibers, whilst at a proportion of 69% fibers there was 40% decrease in strength values. The tensile strength of the composite decreased because of the excessive interaction between the fibers.

Another study on the effect of the length of bagasse fiber on the flexural properties of biodegradable composite was done by Shibata et al. (2005). It is found that different fiber length of the bagasse fibers showed a variation of the flexural modulus and flexural strength of the composite. The flexural modulus of the bagasse fiber below 3mm was found to decrease significantly.

El-Tayeb (2008) studied on the abrasive wear behavior of polymer reinforced with chopped sugarcane bagasse fiber with different lengths (1, 5, 10 mm). Randomly dispersed chopped sugarcane/polyester composite (C-SCRP) was prepared using hand-lay-up method. Despite the good adhesion between fiber and matrix, results of mechanical tests showed poor tensile strength of CSCRP composite. This was attributed to the weak site inside the fiber itself which could not bear the stress transfer from matrix via the fiber. Experimental results of abrasive wear tests revealed that wear of SCRP composite was sensitive to variations of load, fiber length and fiber orientation. C-SCRP composite with 5 mm fiber length offered the highest resistance to material removal compared to the other fiber length used.

On the contrary, Cerqueira et al. (2011) did an investigation on the mechanical behavior of polypropylene reinforced sugarcane bagasse fibers

composites with different volume fraction of the fibers. The sugarcane bagasse fibers (SCB) were pretreated with 10% sulfuric acid solution. For the tensile tests, five specimens were analyzed with dimensions according to the ASTM D 638 standard while for flexural tests, is according to the ASTM D 790 standard. The adopted flexural test was the 3-point at 1/3 points method and a load was applied on the specimen at 2.8 mm min-1 crosshead motion rate. From the results, the highest tensile strength 23 MPa is found to be exhibited by composites with 10 wt% SCB fibers. As a whole, the composites showed an increase of 16% in tensile strength and 51% in the tensile modulus compared to pure resin. While when the fibers were added till 20 wt%, a decreasing trend is noticed for tensile strength. This is due to excessive unnecessary interaction between the fibers and the matrix that lower the tensile strength.

Tensile strength for different grain sizes was observed to be different as well. According to Leite et al. (2004), for bagasse with grain size of 80- 170 meshes, the tensile strength is 36% higher than the bagasse with grain size of that 35-80 meshes. This result can be possibly explained by the grain size that was below the critical length.

Furthermore, surface modification using alkaline surface treatments is introduced to decrease the hydrophilic property of the fiber and causing a better adhesion between the fibers and matrix (R. Syafri, 2011), as many successful attempts have been proved (Mulinari et al., 2010);(Cao et al., 2006);(Cerqueira et al., 2011).

Another study of utilizing of SCB fibers in polyester composites is done by Cao et al. (2006). From this study, SCB fibers are pre-treated with alkali treatment from different concentration (1%, 3%, 5%). The tensile and three-point flexural tests are carried out according to the standard method of glass fiber reinforced plastics (JIS K 7054 and JIS K 7171). Five specimens are developed and tested according to the standard size specified. Improvement of 13% in tensile strength and 14% in flexural strength was reported for 65 wt% of SCB fibers pretreated with 1% of alkali. As for 3% and 5% of alkali pretreatment, the flexural and tensile strength decreases. The results are illustrated in the figure below. This happens

because of greater fibrillation owing to the relatively larger fiber ends available for crack initiation. This would lower the effective stress transfer at the interface and thus affects the mechanical properties.

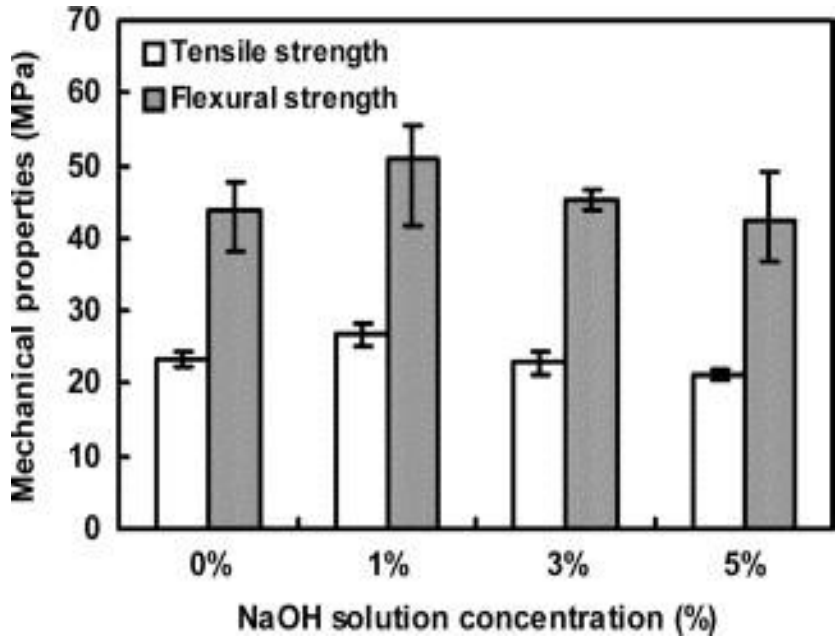


Figure 2. 1 Mechanical properties vs NaOH solution concentration (%) of 65wt% of SCB fibers (Cao et al., 2006)

According to Rout et al. (2001), for the 5% NaOH treated fiber composites, both tensile and flexural strength of the composite will decrease. This happens because of greater fibrillation owing to the relatively larger fiber ends available for crack initiation. This would lower the effective stress transfer at the interface and thus affects the mechanical properties.

2.1.2 Rice husk ash (RHA) particulate

Natural reinforcing material can also be found not just in fibrous form but also in particulate form. Rice husk ash (RHA) is residue which is disposed of by burning at the mill sites and is dumped on a waste land. Utilization of RHA particulate in applications like fillers in cement and fertilizers, catalyst carriers and in the production of pure silica, silica gels, geo-polymer and filled polymers has been reported. RHA is a highly siliceous tough material of high specific properties which is high silica content can be obtained through burning process where the cellulose

and lignin are removed while silica remaining. Depending on the amount of impurities contained and burning conditions, RHA can be divided into two different categories, which are white RHA (WRHA) and black RHA (BRHA) respectively. WRHA can found in grey, purple or white colour. Meanwhile, BRHA consists of mainly carbon due to partially burned (Siddique and Khan (2011)).

Figure 2.2 Rice Husk Ash after burning process

Two forms of silica have been found to dominate in burning (combustion) process. They are lechatelerite which exist in amorphous form and cristobalite that present in crystalline form. Amorphous silica usually possesses high purity, small particle size and high surface area has tremendous potential to be utilized as adsorbent and catalyst support in manifold chemical synthesis (Syed H. Javed (2009)). Besides that, it was reported that amorphous ash possesses greater reinforcing properties because of its higher pozzalonic reactivity (Ayswarya et al. (2012)). RHA is actually a very fine pozzolanic material that can be utilised in cement and concrete in ameliorating the strength and durability while lowering the amount of cement usage and cost required.

Generally, rice husk ash produced from rice husk. The rice milling industry generates bulks of rice husk during milling of paddy which comes from the fields. This rice husk is mostly used as a fuel in the boilers for processing of paddy. Rice husk is also used as a fuel for power generation. Rice husk ash (RHA) is about 25% by weight of rice husk when burnt in boilers. During milling of paddy about 78 % of weight is received as rice, broken rice and bran. While the remaining 22 % of the weight of paddy is received as husk. This husk is used as fuel in the rice mills to

generate steam for the parboiling process. While this husk contains about 75 % organic volatile matter and the balance 25 % of the weight of this husk is converted into ash during the firing process, which is known as rice husk ash (RHA). This RHA in turn contains around 85 % - 90 % amorphous silica. And rice husk ash was obtained by firing rice husk at different temperatures (400, 500, 700 degree Celsius) and thus becoming a carbon neutral green product.

2.1.2.1 Rice husk ash particulate in composites

Research on incorporation of RHA for reinforcing polymer composites are lesser than the studies of RHA add in effects in cement pastes and concrete. Hence, more researches are to be carried out to unveil the plausible modifications and potential of RHA especially in epoxy composite.

A study regarding to the plausibility of utilizing RHA as filler in reinforcing high density polyethylene (HDPE) has been analyzed by Ayswarya et al. (2012). From the study, RHA is observed to possess the highest tensile strength and elongation at break for composites manufactured at a high temperature of550℃. The Young's modulus is increased with higher filler loading and the highest young's modulus is found to be of 2.5 wt% of RHA which is 305 MPa. Another HDPE/RHA composite compatibilized with a compatibilizer or a coupling agent, such as Maleic Anhydride, is also analyzed. The tensile strength is observed to be 18% higher than that of the virgin HDPE while the mechanical properties like breaking elongation and Young's modulus being improved at the same time. The highest Young Modulus for the compatibilized composite is found to be at 320 MPa by RHA with 1.5 wt%.

On the other hand, a comparison of mechanical behaviors of Polypropene composites filled with RHA was done by Turmanova et al. (2008). Young's modulus of all the composites increased with the filler content. The highest young's modulus of the composites is found with composites filled with aerosil and White RHA fillers (WRHA). Composites with 20 %wt of WRHA was found to have 733.5 MPa Young's Modulus while composites with 20 %wt of Aerosil is found to be

having 744.8 MPa Young's Modulus. The pure Polypropene is having only 598 MPa Young's Modulus. The Young's modulus showed an increasing trend with the decrease in filler particulate size. This is due to the larger contact area and better dispersion of the particulates throughout the polymer matrix.

Apart from HDPE and Polypropene, Suwanprateeb and Hatthapanit (2002) conducted a study on RHA based silica as filler for embedding composites in electronic devices using epoxy resin. The comparison is made from black RHA with fused and crystalline silica (commercial fillers) to be embedded into epoxy resin. The weight fraction in this study ranges from 20%- 60%. From this study, RHA- filled epoxy resin has indicated higher mixing viscosity, higher coefficient of thermal expansion (CTE), and higher water absorption percentage. As for the impact strength, RHA-filled composites and other commercial fillers composites are just comparable. While for the tensile strength and elongation at break of silica-filled epoxy are slightly greater than other commercial fillers composites. It is observed that the effectiveness has been reduced due to the residual carbon in the ash. It suggested that controlled combustion decreased the level of carbon content in rice husk ash which may reduce their efficiency as fillers in composites.

Apart from that, Ayswarya et al. (2012) found that tensile strength decreases steadily with the increasing of filler loading. When RHA filler loading increases, the elongation at break decreases. The graph is shown in Figure 2.3 below. This is also due to the poor adhesion of the ash particulates to the matrix observed through SEM analysis.

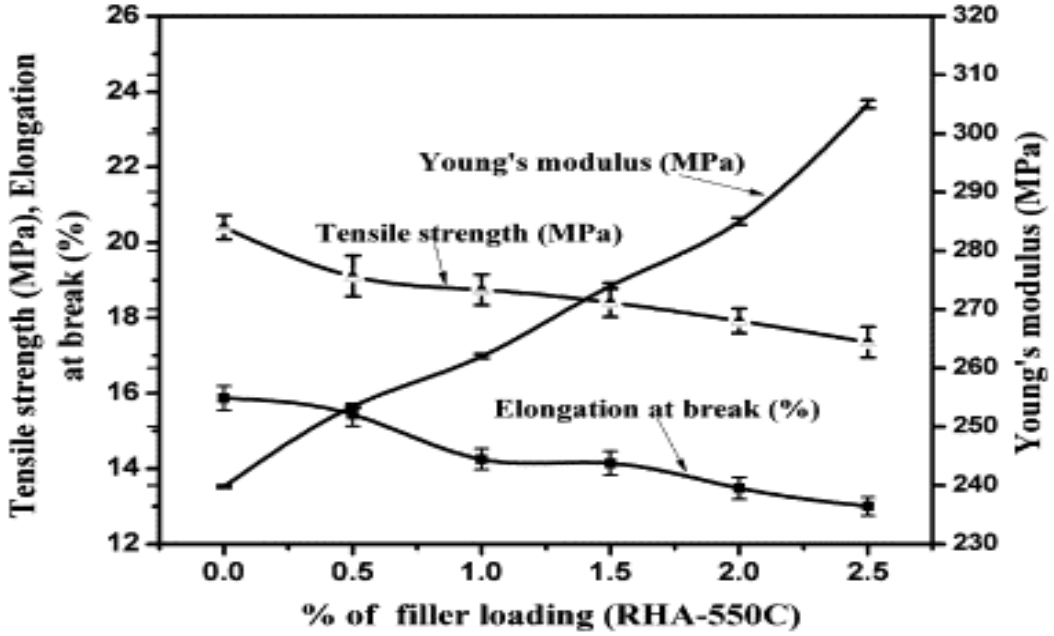


Figure 2.3 Mechanical properties vs filler loading in HDPE

2.2 Synthetic Reinforcing Material

The synthetic reinforcement such as E-glass fibers or Kevlar fibers are commonly found in composites. Low modulus fibers like E-glass fibers are combined with high modulus fibers like carbon fibers to improve the ultimate strain and impact properties of the composites. Apart from fibers, nanoparticles have been utilized as reinforcing phase in composites. Several studies indicate that modulus, strength and toughness can be simultaneously increased with the addition of nano-scale fillers (Chen et al., 2008).

One of the available nano-particulates is silica nanoparticles. It is found to be valuable in the fabrication of composites due to their amorphous structure, SiO_2 content as it achieved more than 99 % purity in most products, and high specific surface area (which leads to the super pozzolanic property) (Bahadori and Hosseini 2012). They are mostly reported in the literature and being extensively used in various applications including electronics, automotive and aerospace industries.

Silica particulates normally exist in fine, white amorphous powder or colloid suspension. Its most significant characteristic is the extremely large surface area and a smooth nonporous surface. The characteristic of silica helps in promoting a strong physical contact when embedded in a polymer matrix (Corandi 2013) and leads to a sharp increase in the interfacial area as compared with the ordinary composites. The interfacial area then creates a significant volume fraction of the interfacial polymer with the properties different from the bulk polymer even at low filler loadings.

Utilization of nano-particulates is also known as additives. Addition of nano-particulates into the matrix helps to improve adhesion and mechanical properties of composites. Utilization of nanoparticles has improved the stiffness of polymers (Dittenber et al., 2012).

Lignocellulosic material along with mineral filler (nano-SiO_2) can be successfully utilized to produce fiber-reinforced composites with better physical and mechanical properties. Higher nano-SiO_2 content had improved the mechanical properties of the composites reinforced by lignocellulosic material. According to Deka et al. (2011), the incorporation of nanoparticles has improved the hardness value due to the filling role of nano-SiO_2 particles and their high specific surface. Besides, due to the filling gaps of nano-SiO_2 particles, the water absorption of composites was less than 0.35 % even in longer duration of moisture absorption.

Fakirov, S. (2009), did a research for nanocomposites that are filled with polystyrene (PS), Polypropylene (PP) or poly(methyl methacrylate) (PMMA) grafted nanoparticles (SiO_2). There is a substantial rise in the strength of SiO_2-g-PS/PP composites at a SiO_2 content as low as 0.65 vol%, then the strength remains almost unchanged with further addition of the filler. A similar behavior can be observed in the case of SiO_2-g-PMMA/PP, except that a drop in strength occurs when the filler content exceeded 1.96 vol%. The decrease in tensile strength of SiO_2-g-PMMA/PP above 1.96 vol% can be understood by a change in the dispersion status of the fillers. It is believed that a higher filler loading might be detrimental to its uniform dispersion in the polymer matrix and lead to the

formation of agglomerates. Thus, affecting the tensile strength of the nanocomposite.

2.2.1 Synthetic fibers-reinforced composite

According to Joseph et al. (1999), for composites reinforced with short fibers, there is a critical length of fiber necessary if maximum resistance is to be achieved. If the length of the fiber employed as reinforcement is shorter than the critical length, the fiber will be loosened from the matrix and the composite will break at low tensions. The results indicate that the properties increased with the length of the fibers and that the critical length lies in the range of 35-45mm.

Another study regarding the tensile properties of polypropylene (PP) reinforced with short glass fibers (SGF) and short carbon fibers (SCF) was done by Fu et al. (2000). Both specimens were prepared with extrusion compounding and injection molding techniques. The tensile properties of specimens were determined from taking 10 samples for each composition with a Zwick 1456 testing machine, according to the standard, DIN 53455. The addition of both glass and carbon fibers of size 50mm, effectively improves the ultimate strength as the ultimate strength of pure PP matrix is 31.6 MPa, while SCF/PP is 59.5 MPa and SGF/PP is 51 MPa. Moreover, it can be seen that the strengths of SCF/PP composites are higher than those of SGF/PP composites. This is because carbon fibers have a much higher strength than glass fibers.

Zhang et al. (2007) investigated the influence of effect of fiber length on the wear resistance of short carbon fiber reinforced epoxy composites. In this work, the sliding performance of composite specimens against polished steel counterparts under dry conditions was studied by using both block-on-ring and a pin-on-disk apparatus. The flexural modulus and strength were investigated under a three-point-bending approach according to DIN-ISO-178, where five specimens were cut into a dimension of $100 \times 10 \times 4$ mm^3. The test speed was kept constant at 1 mm/min. It was found that composites with longer short carbon fiber (SCF) (nominal length = 4mm) exhibited better wear resistance than those with shorter SCF

(nominal length = 0.9mm). It was also found that within the range of critical fiber length, a longer fiber length usually corresponds to a higher stiffness of the composite, and vice versa.

2.2.2 Synthetic particulate- reinforced composites

Apart from fibers, there are also other fillers such as particulates being used in reinforcement of composites. Nanoparticles have been utilized as reinforcing phase in composites. Several studies indicate that modulus, strength and toughness can be simultaneously increased with the addition of Nano-scale fillers (Chen et al., 2008).

Chen et al. (2008) have investigated the thermal and mechanical properties as a function of the silica percentage with spherical silica particulates of size 12-nm. Characterization techniques utilized in order to investigate the morphology are transmission electron microscopy (TEM) and ultra-small-angle X-ray scattering (USAXS). It is found that nanoparticles can be dispersed with minimal aggregation when the amount used is up to 25 wt%. It is reported that there is an increase of 25% in tensile modulus and 30% in fracture toughness for samples less than 10 wt% of silica.

Another study from Alamri and Low (2012) found that the flexural strength of the epoxy composites filled with nanosilicon carbide (n-SiC) increased by 21.5% due to the presence of 1 % n-SiC particulates. The n-SiC nanocomposites were prepared by mixing the epoxy resin with three weight percentages (1%, 3%, 5%) with a high speed mechanical mixer for 10 minutes. For the flexural modulus, addition of 1% of n-SiC cause an increase of 83% of flexural modulus of the epoxy system. The enhancement in these properties is ascribed to the good dispersion of n-SiC particulates into the matrix. However, when the n-SiC loading is increased to 3% and 5%, the flexural strength decrease to values less than that of the pure epoxy. This happens because of the poor dispersion of particulates inside the matrix. The agglomerations act as stress concentrators which result in reduction of flexural strength.

2.3 Hybrid composites

In sections 2.1 and 2.2 the composites which are manufactured in combination of one kind of synthetic or natural fiber and particulates as fillers are discussed. However, there are shortcomings in areas such as the agglomeration of fibers and particulates, fibers pull-out and poor wettability of the resin which result in poor mechanical properties when the fillers exceed certain volume fractions. Hence, hybrid composites are developed in order to achieve better mechanical properties by preventing above mentioned issues of fibers and particulates with the resins.

Alamri and Low (2012) investigated the mechanical properties of the epoxy hybrid composites filled with Recycled Cellulose Fibers (RCF) and nanosilicon carbide (n-SiC). The RCF sheets are dried under 70˚C for 60 minutes before the sheets are soaked into the mixture of epoxy and n-SiC particulates. The combination of these two kinds of fillers, n-SiC and RCF have resulted in a major improvement in the values of flexural strength and modulus of the composites. From Figure 2.4 below, the chart showed the significant difference of the mechanical properties (flexural properties) between unfilled composites and RCF filled composites.

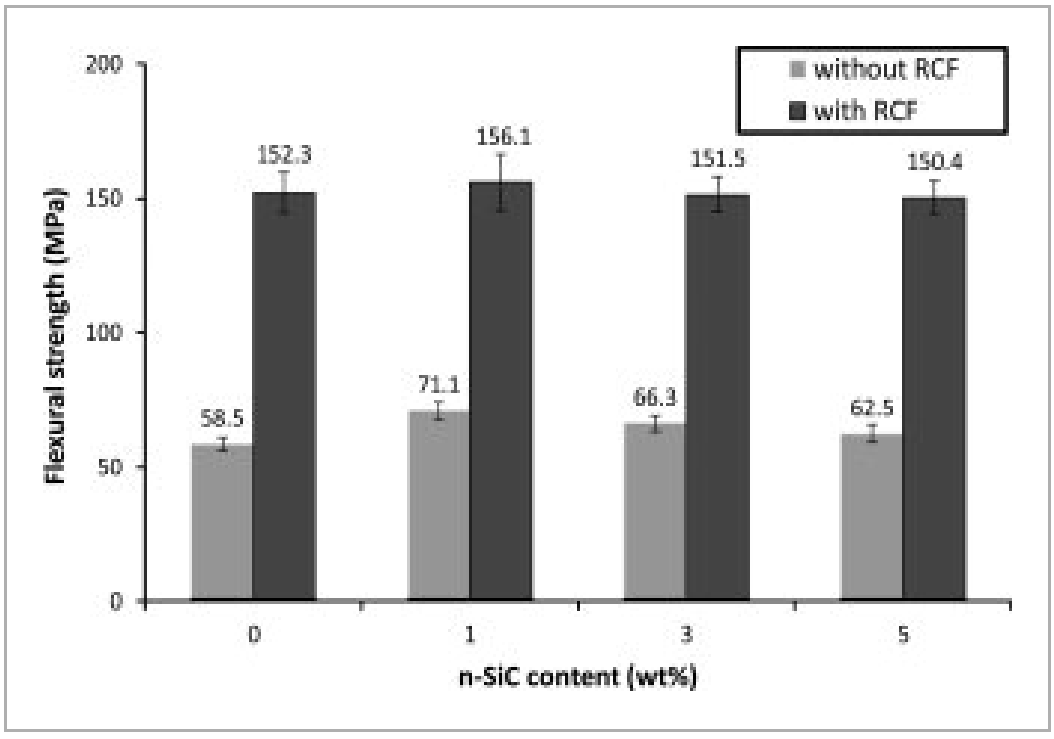


Figure 2.4 Flexural strength against n-SiC (wt %)(Alamri & Low, 2012a)

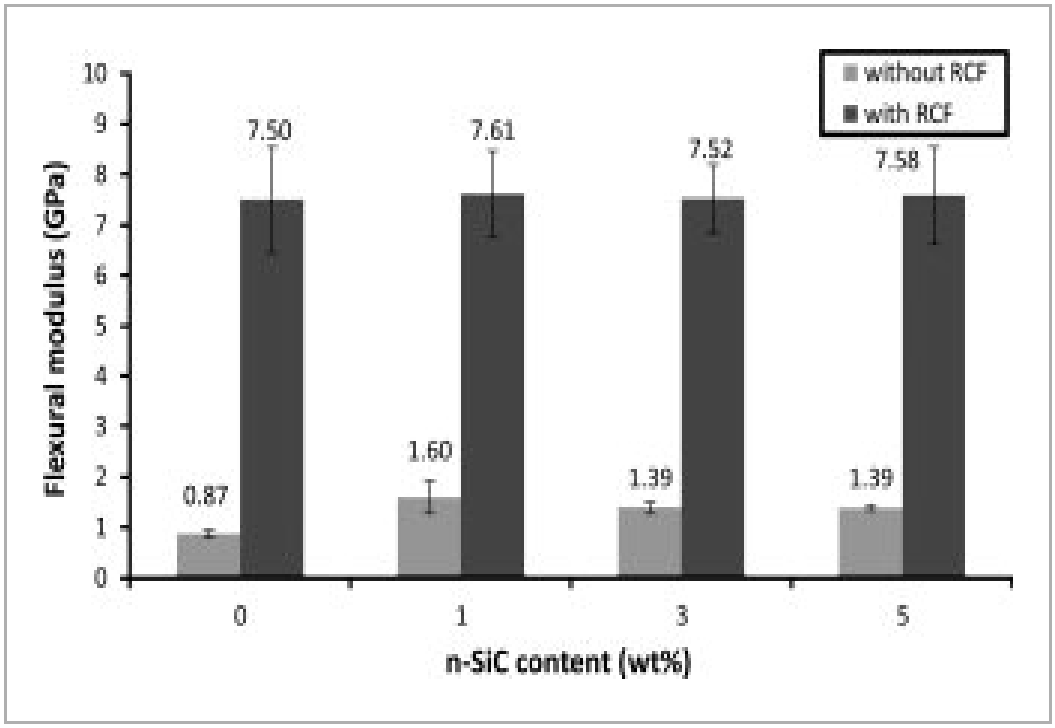


Figure 2.5 Flexural modulus against n-SiC (wt %)(Alamri & Low, 2012a)

While another study on silica and rice husk ash hybrid composite of white rice husk ash (WRHA) in polypropylene (PP) has been carried out by Ismail et al. (2003). Tensile tests were carried out according to ASTM D412 where 2mm dumbbell specimens were cut from the molded sheet. Furthermore, flexural tests were carried out according to ASTM D790. From these experiments tensile strength was found higher for composites with higher silica/WRHA volume fractions. For the silica/ WRHA ratio of 40:0, the tensile strength was found to be 28MPa while when the ratio is 0:40, the tensile strength was found to be 22.5 MPa. When WRHA content is higher in the silica/WRHA weight ratio, the tensile modulus and the flexural modulus of the composite also increased. For the silica/ WRHA ratio of 40:0, the flexural modulus was found to be 2340 MPa and for 0:40, the flexural modulus was about 2500 MPa.

A study of tensile properties of composite materials of SCB fibers, glass fibers in epoxy matrix is done by Tewari et al. (2012). Although there are improvements of mechanical properties such as modulus of elasticity, E, and also percentage of elongation, there are still decrements of ultimate strength of the composite when bagasse fibers were introduced into the epoxy matrix. It was

observed from SEM images that the cause of the deterioration was the binding of fibers with the epoxy matrix.

Another experimental study on the mechanical properties of natural fibers reinforced hybrid composites has also been conducted by Parandaman and Jayaraman (2015). Two lightweight composites materials were developed, one with linear pattern and another with a chopped pattern of bagasse and coir fibers reinforcements. The tensile strength of the composite was found to be relying on the strength and length of the fiber and also the fiber-matrix interaction. From the results, it was observed that the best ultimate tensile strength, *UTS*, of the chopped fiber composite was 12.5 N/mm^2 and was higher than the continuous fiber composite which was found to be 11 N/mm^2. For the hardness test which was conducted by Rockwell Hardness method, it was found that the chopped fiber composite also possessed higher Rockwell hardness values of 49 compared to the continuous fiber composite of 37.3.

While on the other hand, Braga and Magalhaes (2015) conducted the investigation about the mechanical and thermal properties for jute fiber and glass fiber reinforced epoxy hybrid composites. The mechanical properties increased with more proportions of glass fibers. Table1 below summarized the percentage by mass of sample and mechanical properties of the samples. The tensile strength of the composite C2 increased by 68.70% to Composite C1 and further increase by 92.01% to composite C3 in relation to the composite C1. The flexural strength did not have a significant increase, which was only by 1.99% to C2 composite and by 2.24% to C3 composite in relation to C1 composite.

Table 2.1 Percentage by mass of samples and respective mechanical properties (Braga & Magalhaes, 2015)

Symbol	**Percentage by mass of samples**			**Mechanical Properties**		
	Epoxy resin (% in mass)	**Jute fiber (% in mass)**	**Glass fiber (% in mass)**	**Tensile Strength (MPa)**	**Flexural Strength (MPa)**	**Flexural Modulus (MPa)**
C1	69	31	0	29.52	28.18	1249.92
C2	68	25	7	49.80	28.74	1189.18
C3	64	18	19	56.58	28.81	1830.68

Another study from Raghavendra et al. (2014) is regarding the comparison of the properties of jute fiber and E-glass fiber in epoxy matrix. Thermal analysis is the concept that chastely replicates the reactions that occur at the molecular level of the materials. From the curve gained from the thermogravimetric analysis (TGA), it is found that jute fiber is less compared with the neat epoxy matrix. Which means that the jute fiber is less in terms of thermal stability. In terms of tensile strength, comparing the jute fibers reinforced composite with the glass fibers reinforced composite, the jute fiber reinforced composites gives 55% strength of glass fiber. While for the flexural strength, jute fiber composite gives 61% strength of the glass fiber composites. The results obtained showed that natural fiber reinforced composites are comparable with conventional fiber composites in term of mechanical properties.

Gopinath et al. (2014) have conducted a study regarding jute fibers reinforced with polyester and epoxy resin. The type of jute fiber that is used is olitorious jute and the jute are treated separately with 5% and 10% NaOH solution for 24 hours. The specimen of composite are prepared according to ASTM standard for tensile testing and flexural testing (ASTM D3039 and ASTM 790). It was reported that the jute-fiber composite treated with 5% NaOH solution showed better mechanical performances compared with those which are treated with 10% NaOH solution. The tensile strength was 18.67% higher for the jute-epoxy composite while 16.67% higher for the jute-polyester composite. The jute-epoxy composite was about 20% higher in flexural strength, compared to fibers treated with 10% NaOH solution while jute-polyester composite was 15% higher. This once again proven that the effective stress transfer at the interface affected the mechanical properties by the fibrillation that was too extreme.

2.4 Epoxy Resin

Epoxy is one of the thermosets polymers that have covalent bonds linking the polymer chains in three dimensions. These links prevent the chains from sliding past one another resulting in a higher modulus and improved creep resistance.

Epoxy resin is converted from liquid state to a hard rigid solid by chemical cross-linking through a curing process which involves the application of heat and the addition of curing agent, which is also known as hardeners. A tightly bound three-dimensional network structure is molded in the resin once curing process is done. The resin cannot be melted, reshaped or reprocessed by heating. Therefore, the impregnation process followed by the shaping and solidification should be done before the resin begins to cure during composite manufacturing (Kathiresen 2004).

Increased resin/fiber adhesion is generally derived from both the resin's chemistry and its compatibility with the chemical surface treatments applied to fibers. Polyesters, vinylesters, and epoxies are accounts for 90% of all thermosetting resin systems used in structural composites (Mohanty et al. 2002). While epoxy resin has the higher percentage of Young modulus compared to polyester and vinylester. The adhesive properties of epoxy can help laminates to achieve higher micro-cracking strains (Penczek 2005). The strain that a laminate can reach before micro cracking depends strongly on the toughness and adhesive properties of the resin system. As the ultimate strength of a laminate in tension is linked to the strength of the fibers, these resin micro-cracks do not immediately reduce the ultimate properties of the laminate. However, compared to an uncracked laminate, the micro-cracked laminate will absorb more water in a moist environment. This will lead to an increase in weight, moisture attack on the resin, loss of stiffness and lead to an eventual deterioration of ultimate properties.

2.5 Deficiency of the natural fibers/ particulates- reinforced composites.

In the previous sections, the natural and synthetic fibers and particulates reinforced composites are reviewed. There are still drawbacks of these composites mainly caused by the incompatibility between fibers and matrices, tendency to form aggregates during processing causing poor interfacial adhesion (Cerqueira et al., 2011). Hence, the key deficiencies of natural fibers-reinforced composites are summarized in this section.

2.5.1. Interfacial adhesion

According to Huang et al. (2012), the chemical composition of sugarcane bagasse is composed of three main components which include hemicellulose, lignin and cellulose. The cellulose component is comprised of units of an hydro-d-glucose, with each unit containing three hydroxyl groups (Huang et al., 2012). Thus, since fiber surfaces have waxes and other non-cellulosic substances such as hemi-cellulose, lignin and pectin, which will create poor adhesion between matrix and fibers. Furthermore, the tensile strength and the fracture patterns are highly dependent on the interfacial adhesion between the phases present. Hence, if the adhesion is poor, there is a strong probability of creating a flaw that would lead to breakage of the material in the interfacial region. The other drawback is the high moisture absorption and incompatibility between the natural fibers and matrix (Ma et al., 2005). Due to the plant chemical structure, all plant-derived cellulose fibers are polar and hydrophilic in nature. Non-cellulosic components such as hemicelluloses and pectin are the most hydrophilic as they contain many accessible hydroxyl (OH) and carboxylic acid groups, which are active sites for the absorption of water (Lilholt et al. 2000). Hydroxyl groups (--OH) in the main backbone chain of a resin provide sites for hydrogen bonding to the surface of the natural fibers, which contain many hydroxyl groups in their chemical structure (Ray and Rout, 2005). On the contrary, the epoxy resin is hydrophobic, having no hydroxyl group in its backbone chain generally has the weakest bonding. Hydrophobicity is one of the properties of a molecule which relates to the repulsive behavior between non-polar substances and water (Gilfillan et al., 2012).

2.5.2. Particulate agglomeration

As a whole, the following defects found in natural particulates reinforced composites are the main drawbacks of utilizing these natural fillers in composites. When the particulates added to the matrix has reached a certain amount, the particulates may agglomerate, and this could weaken the adhesion strength between the matrix and the filler. The incompatibility between the natural fibers and the polymer matrix results from the different characteristics of the hydrophilic fibers and hydrophobic matrix cause a tendency to form aggregates. These

agglomerations may become stress concentrators, which are the cause of reduction in flexural strength. When excessive fibers are embedded in the composites, unnecessary interaction between the fibers will decrease the tensile strength of the composite. Furthermore, weak bonding between the fiber-matrix interface and insufficient fiber dispersion could lead to poor stress transfer, which results in a nonhomogeneous mixture (Gilfillan et al., 2012). As discussed in former section, the agglomeration could occur while there is insufficient of fiber dispersion which the epoxy resin areas are weak while bagasse fiber areas (agglomeration) are susceptible to micro cracking (Painchaud et al. 2006). The improvement of the mechanical properties of a composite material based on natural fibers depends on the strength of the bond between the fibers and the polymer matrix (Gilfillan et al., 2012).

2.5.2. Voids

Beside the agglomeration of fibers and particulates, the voids present in the matrix after the combination of resin and fillers are also one of the causes of composites with poor mechanical properties. The absence of any voids around the fibers may show a good adhesion between fibers and matrix.

2.6 Problem statement

Currently, there are several significant challenges faced in the application of natural fiber-polymer composites including poor interfacial adhesion and incompatibility between the natural fibers and matrix. Utilization of longer fibers can further increase their tendency to agglomerate. The efficiency of reinforcement could be reduced due to poor fiber dispersion as fiber are entangled during the mixing process. As the fiber content increased in the matrix, the bonding between the fiber and the matrix start to deteriorate when the fibers overlap in the matrix. Besides, poor stress transfer due to poor interfacial bonding between them causes agglomeration to form and thus affects the mechanical properties. Hence, this study will address the probability of enhancing interfacial properties between the fiber, the particulates and the matrix. This study will also minimize the agglomeration problems of the particulates and develop the bio-composite with enhanced mechanical properties.

From the research challenges that were addressed in the discussion above, the research questions are formed and listed below:

1) How to improve the mechanical performance of epoxy composite?
2) What is the consequence of adding fillers with different forms (ie: particulate and fibrous) into epoxy matrix?
3) What is the consequence of using the mostly natural fillers in the composite?

2.7 Objectives

The primary objectives of this project is to develop a bio-composite material that is sustainable and commercially feasible. The specific objectives are as follows:

1) To analyze the mechanical properties (tensile and flexural properties) of bio-composite that consist of Sugarcane bagasse (SCB) fibers, Rice Husk Ash (RHA) particulates, nanosilica particulates and epoxy matrix to produce a hybrid bio-composite material with enhanced mechanical performances. Hybrid combination of composite that will be produce consist of:

a) Manifold weight percentages (1%, 2.5%,5%, 7.5%) of SCB fillers in epoxy matrix.

b) Manifold weight percentages (1%, 2.5% 5% 7.5%) of RHA particulates in epoxy matrix.

c) Various hybrid combinations of SCB fibers, RHA particulates and nanosilica in epoxy matrix.

2) To investigate the morphological properties including the microstructure and fracture surface of the hybrid bio-composite material. Identify the

effects of microstructure of the composites to the mechanical performances of the composites.

2.8 Hypothesis

The combination of Sugarcane bagasse (SCB) that exists in fibers form, rice husk ash (RHA) as particulates and nanosilica have not used before for the purpose of generating a hybrid composite material with enhanced mechanical performances (tensile and flexural properties). Furthermore, this combination has yet to be incorporated into the polymeric matrix of epoxy resin in past research to understand the interrelational behaviors while discovering the enhanced mechanical properties being introduced. While some research has been carried out on using different weightage of fiber to improve the mechanical performances of the resulting composites, there has been no detailed investigation of using the sugarcane bagasse short fibers and particulates to enhance mechanical performances (tensile and flexural properties). Avoiding the agglomeration of particulates in the composites produced while fulfilling the voids present in the composites will further improve the mechanical properties of the desired composites. A schematic diagram that depicts all the fillers of the composite is shown in Figure 2.5.

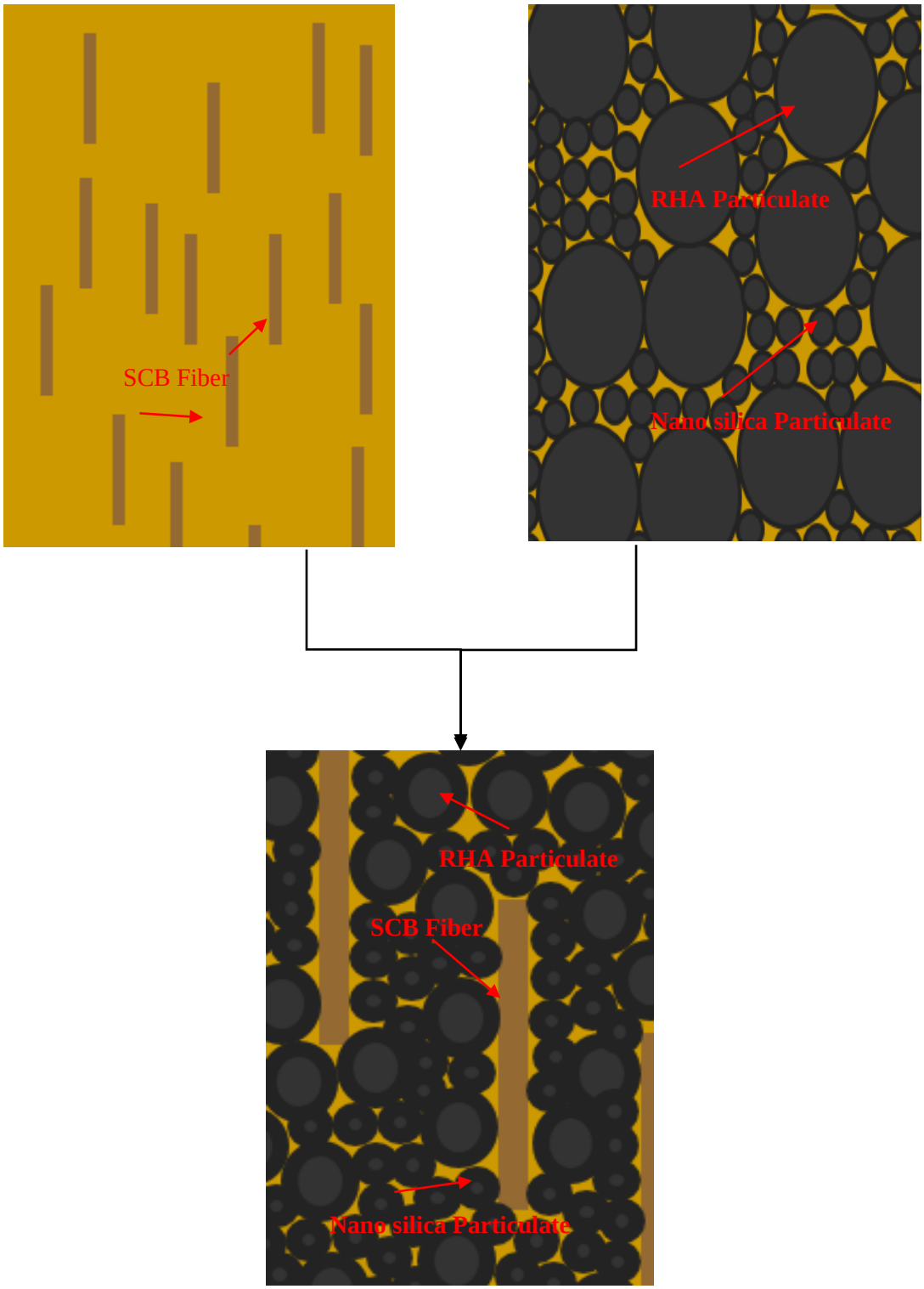


Figure 2.6 Schematic diagram of the proposed composite

2.9 Scientific merit and significance of research

Due to the escalating problem arising from exploiting the natural resources, the utilization of natural fibers, such as sugarcane bagasse fibers (SCB) is favored in the manufacturing of composite. However, SCB alone as the sole filler is proven to be insufficient for enhancing the mechanical properties of composite. Hence, several fillers such as rice husk ash (RHA) and nanosilica are chosen to be the reinforcing medium to the primary medium, epoxy matrix.

Scientific significant of the research includes:

I) A novel bio-composite material with the combination of SCB fibers, Rice husk ash (RHA) and nanosilica particulates being incorporated into the epoxy resin. When SCB fibers are embedded into the epoxy in randomly distributed form, there are voids formed that are caused by the overlapping of fibers in the matrix. Hence, RHA in this case, will serve as the micro-filler, in order to fill the spaces in between the fibers. While nanosilica serve as the nano-filler filling the nano-spaces in the composite system.

II) Agglomeration and uneven distribution of reinforcement in matrix often occur when too much fillers of one kind are embedded into the resin. Hence, when certain amount of nanosilica and RHA is introduced into the resin, it is highly anticipated that the micro-voids in the epoxy resin can be reduced, while agglomeration can be minimized too.

2.10 Summary of Chapter

The most recent related literatures regarding the synthetic fibers and particulates reinforcement in composite materials are reviewed in this chapter. For natural fiber composites, sugarcane bagasse fibers and rice husk ash particulates reinforcement are mainly considered. The combination of Sugarcane bagasse (SCB) that exists in fibers form, rice husk ash (RHA) as particulates and nano-silica

have not used before for the purpose of generating a hybrid composite material with enhanced mechanical properties (tensile and flexural properties). Furthermore, this combination has yet to be incorporated into the polymeric matrix of epoxy resin in past research to understand the interrelational behaviors while discovering the enhanced mechanical properties being introduced. As the size of the rice husk ash particulates is in micron while the size of nanosilica is in nano-size, the nanosilica is able to fill the smaller voids in the resin while rice husk ash particulates will fill the bigger voids in the resin. This will avoid the agglomeration of particulates in the composites produced while fulfilling the voids present in the composites. This will further improve the mechanical properties of the desired composites.

CHAPTER 3
RESEARCH METHODOLOGY

3.1 Background

In this chapter, the research methodology is proposed. In this study, Epoxy resin (Miracast 1517 A/B) is reinforced with three different fillers, namely sugarcane bagasse (SCB), rice husk ash (RHA) and nanosilica. Different forms of SCB namely particulate and fibrous form of a range of weights are reinforced into epoxy matrix to evaluate the mechanical performance of the composites. Furthermore, RHA and nanosilica with different combination of weight percentages are also reinforced into the epoxy matrix to explore its mechanical behavior.

The research method includes the surface treatment of the natural fillers, preparation of the composites consisting of epoxy matrix, SCB and RHA fillers, and nanosilica of different weight percentages. Subsequently the methodology discusses the methods of mechanical properties testing and morphological analysis. Figure 3.1.1 represents the flowchart of the overall methodology in this research work.

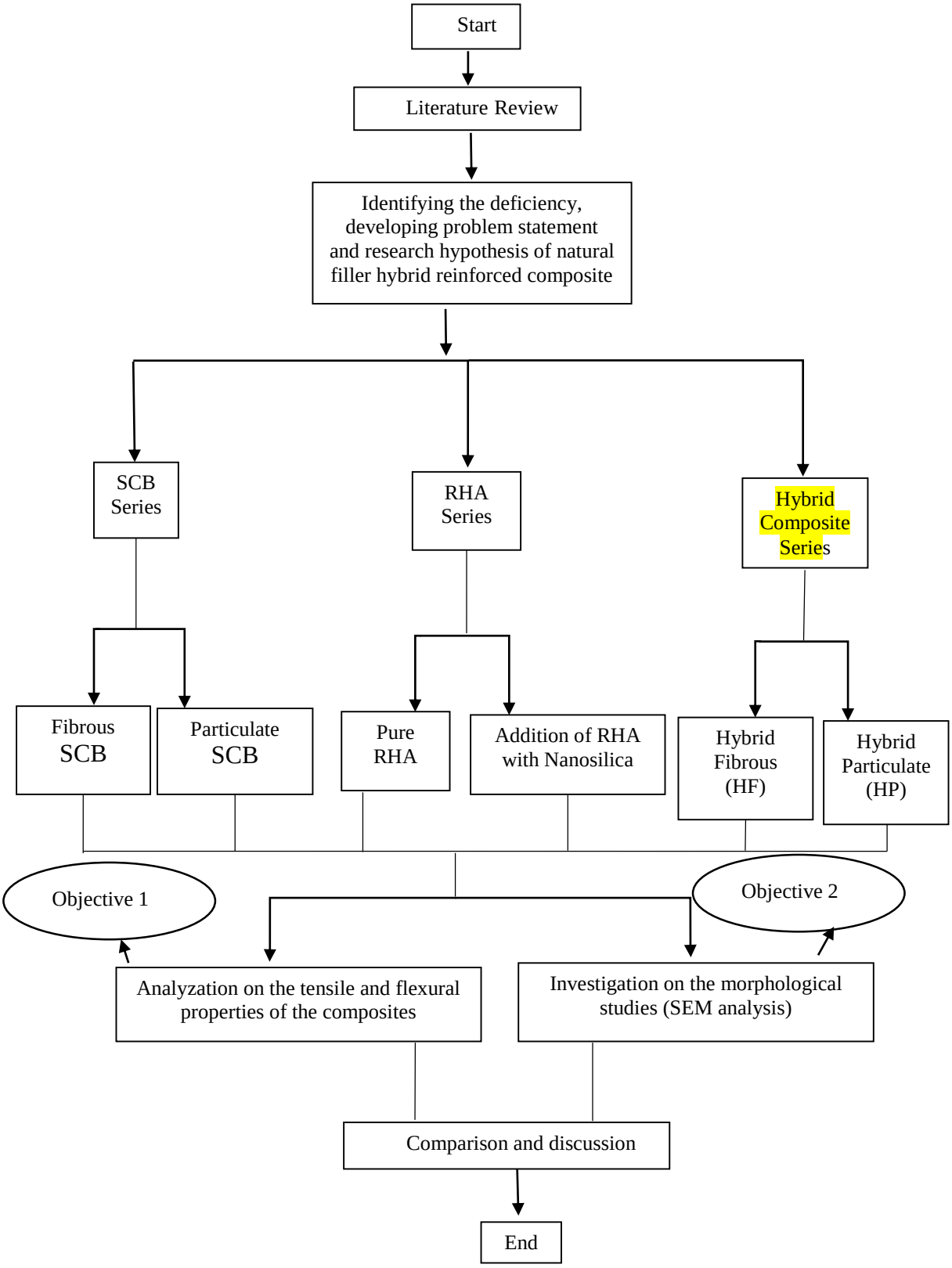


Figure 3.1.1 Flowchart of the methodology

3.2 Composite fabrication method

The fabrication method of the proposed composite which includes the list of materials, properties of epoxy, preparation of materials are discussed in this section. Table 3.2.1 shows the list of materials and Table 3.2.2 shows the properties of Epoxy Matrix and Hardener used in this research work.

Table 3.2.1 List of materials

No.	Materials	Function	Supplier/Manufacturer
1.	Sugarcane Bagasse Fibers	Reinforcing Filler Material	Local Suppliers of Sugarcane Juice
2	Rice Husk Ash	Reinforcing Filler Material	LLH Biomass Sdn Bhd
3.	Miracast 1517 A	Matrix Material	Miracon Sdn Bhd
4.	Miracast 1517 B	Catalyst	Miracon Sdn Bhd
5.	Sodium Hydroxide Pellets	Alkaline Surface Treatment	Peter Scientific and Electronic Enterprise
6	Meguiar Maximum Mold Release Wax	Parting Agent between Layers of Matrix and the Mold Surface	Amazon
7	Nanosilica (nS)	Nanoparticles filler	Sigma Aldrich

Table 3.2.2 Properties of epoxy matrix

	Properties	Test Methods	Units	Results
Miracast 1517 A	Colour Viscosity, 25˚C Specific Gravity	Visual Brookfield ISO 2811	- CPS g/cm^3	Clear 2000-4000 1.2-1.3
Miracast 1517 B	Colour Viscosity, 25˚C Specific Gravity	Visual Brookfield ISO 2811	- CPS g/cm^3	Clear 25-75 0.9-1.1
Miracast 1517 A/B	Mixing ratio Mix viscosity, 25˚C Density Pot life, 25˚C	- Brookfield ISO 2811 -	pbw CPS g/cm^3 minutes	100:30 Approx. 400 Approx. 1.13 20-30
Physical Properties	Hardness Tg	ASTM D2240 ISO 11357-2	Shore D ˚C	80 80

3.2.1 Preparation of surface treatment (alkali treatment)

The preparation and procedures of alkaline surface treatment on the SCB short fibers and particulates are discussed in this section.

Determination of the mass for NaOH (sodium hydroxide) pellets

The Weight Percentage formulation will be utilized to fix the required mass of NaOH pellets in order to form the 2 wt% concentration of NaOH solution.

The Weight/Volume Percentage formula is provided below:

$$Concentration\ (wt\ \%) = \frac{Mass\ of\ Solute\ (g)}{Volume\ of\ Solution\ (ml)} \times 100\%$$

The mass of the NaOH pellets required is tabulated in Table 3.2.3.

Table 3.2.3: Determination of the mass of sodium hydroxide pellets required

Concentration of NaOH solution	Mass of Solute Required
2 wt%	$2\% = \frac{Mass\ of\ Solute\ (g)}{500\ ml} \times 100\%$ $Mass\ of\ Solute\ (g) = 2\% \times \frac{500}{100\%}$ $= 10g$

Preparation of NaOH solution of 2% concentration (alkali treatment)

The mass of the NaOH pellets was identified by utilizing the weighing balance equipment. Then, it is calibrated to zero reading to prevent systematic errors. The 500 ml beakers and a 600 ml volumetric flask were rinsed thoroughly with distilled water to eliminate the impurities. Next, NaOH pellets were placed accordingly into the disposable cup while confirming an accurate reading of 10 g on the display monitor of the equipment. The measured NaOH pellets were then placed into the 600 ml volumetric flask. Distilled water was then poured slowly into the volumetric flask, up to 100 ml. The mixture of 10 g NaOH pellets and the 100 ml volume of distilled water were stirred using electronic stirrer until the aqueous solution became homogenous, as illustrated in Figure 3.2.1

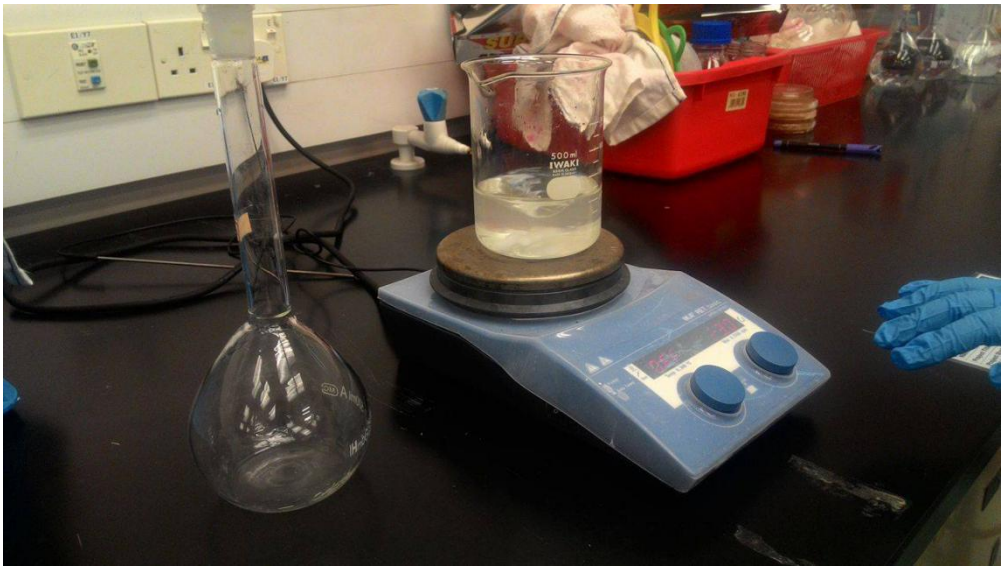


Figure 3.2.1 Stirring of sodium hydroxide pellets and distilled water using the electronic stirrer

Procedures for treating the SCB fibers in length and particles using NaOH solution

Sugarcane bagasse fibers (SCB) are prepared according to the desired length for fibrous form and desired size for particulate form. For fibrous form, the dried inner pith of the sugarcane fibers was first extracted manually and cut into the length of 5-10 mm accordingly using a pair of scissors. Extreme care had been taken to avoid the damages of SCB fibers as they were attached and surrounded by thick layers of piths. The fibers were placed in air-tight containers to ensure they are kept in dry and cool place. As for particulate form, Endecotts EFL 2000 Test Sieve Shaker was used to sieve grounded bagasse fibers into desired sizes of particulate. Furthermore, manually sieving was also used to sieve the smallest size of particles as the sieve was easily clogged due to tiny holes of the sieve shaker. The 600 µm sieves is used to sieve the sugarcane bagasse particulates. The 0.600 mm sieve was covered with a lid and the locking handle was sealed tightly onto the spiral pole by using the black round handle. The preparation of SCB is shown in Figure 3.2.2 and Figure 3.2.3 below.

Figure 3.2.2 Collection of sugarcane bagasse, removing of outer rind and cleaning of inner pith (SCB)

Figure 3.2.3 Sieved sugarcane bagasse particulate that is having a size of 0.6mm

SCB short fibers and particulates were measured by weighing balance. The bagasse short fibers and particulates were place into separate containers. The NaOH solution of 2 wt% concentration was transferred into both containers. The colour of the mixture was transformed from white to yellow, signifying that deoxidization of the SCB fibers had taken place. This is shown in Figure 3.2.4. The mixtures of the NaOH solution and the fillers were stirred using a spatula. The SCB particulates were left to be soaked in the NaOH solution for 24 hours at room temperature. After 24 hours, the alkaline treated SCB particles were washed and then immersed in the distilled water to get rid of any excessive NaOH. The alkali treated SCB particulates were then put on an aluminium foil in a tray and the fibers were distributed evenly on the surface of the aluminium foil. The treated SCB were then dried in an oven for 150°C for a duration of 4 hours, as shown in Figure 3.2.5. Afterward, the dried SCB fibers were kept in separate air-tight containers, one for bagasse particles and another for short fibers.

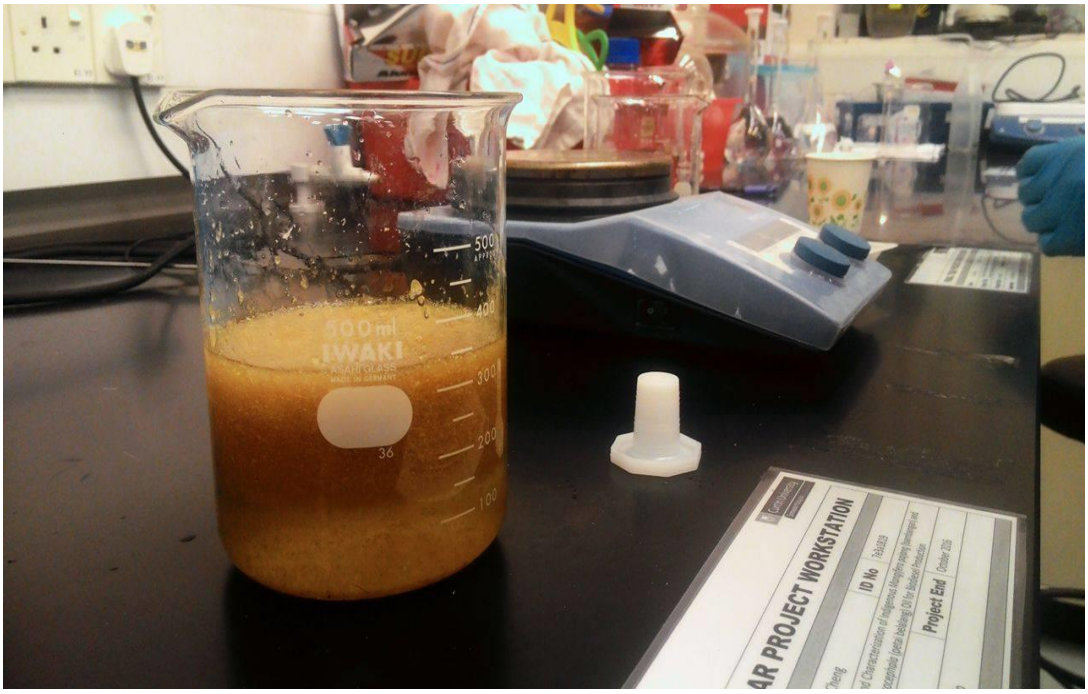


Figure 3.2.4 Bagasse particles in NaOH solution

Figure 3.2.5 The treated fibers were dried in an oven

Preparation of rice husk ash (particulate)

Rice Husk Ash is acquired from LLH Biomass Sdn. Bhd. The raw rice husk ash is separated from dirt and impurities by a sieve. It is then sieved to 75 µm, stored in a dried container and sealed properly.

3.2.2 Preparation of different filler loading

Determination of the mass of fillers required to obtain different weightage of composite

The different filler loading of sugarcane bagasse short fibers and particles are calculated by the weight percent (w/v) formulation as follows:

$$\text{Mass of filler (g)} = \frac{Resin\ and\ Hardener\ Required\ (g)}{100\%} \times 26\text{g (mass of epoxy+hardener)}$$

The weight percent (w/v) formulation shown as above was used to find the masses of sugarcane bagasse fibers (g) filler loading required to mix with 20g of epoxy and 6g

of hardener. Accordingly, the calculated masses of sugarcane bagasse filler are 0.26g, 0.78g, 1.3g, and 1.82g for the required filler loading of 1wt%, 3wt%, 5wt% and 7wt% respectively. The mass of filler for RHA, nanosilica and hybrid series are identical to SCB series, thus it is not shown separately. The experimental setup of SCB, RHA and nanosilica particulates combinations of different weight percentages is shown in Table 3.2.4

Table 3.2.4 Experimental setups with different fillers and weight percentages

SCB Series Set 1: NaOH Treated SCB fibrous Set 2: NaOH Treated SCB particulates				
1	1% SCB	2.5% SCB	5% SCB	7.5% SCB
2	1% SCB	2.5% SCB	5% SCB	7.5% SCB
RHA series Set 3: Pure RHA particulates Set 4: Nanosilica (Ns) + RHA (NRHA) particulates				
3	1% RHA	2.5% RHA	5% RHA	7.5% RHA
4	1% Nanosilica (Ns)	1% Ns + 1.5% RHA	1% Ns + 4% RHA	1% Ns + 6.5% RHA
Hybrid Series				
HF250	1% Ns + 1% RHA + 1% SCB (fibrous)			
HP250	1% Ns + 1% RHA + 1% SCB (particulate)			
HF750	1% Ns + 3% RHA + 3% SCB (fibrous)			
HP750	1% Ns + 3% RHA + 3% SCB (particulate)			

Preparation of the composites of different weightage

Molds are prepared according to ASTM standards. Different weight percentage of fillers were used in this experiment. The formulation of different weight percentage is shown in the previous section. Epoxy resin and its hardener were poured into two separate disposable cups. An empty disposable cup was placed on the weighing balance equipment and was calibrated to prevent zero error. For the case of 1wt%

SCB, 18.02 g of epoxy was poured into the cup and was also calibrated to zero reading using the weighing balance. 7.72 g of hardener was weighted and poured into the same cup. The mixture of the epoxy and hardener catalyst was then stirred manually for 3 minutes. Alkaline treated SCB fiber of 0.26 g was added into the mixture prepared earlier. Meguiars Maximum mold release agent was applied on the molds as a parting agent between layers of the matrix and the mold surface. The mold release agent allows the hardened composite to be taken off from the mold without harming it. The mold release wax is shown in Figure 3.2.6. The mixture was then poured into the tensile molds by using hand-lay method. The composite was then placed into the desiccator, and vacuum gas pump was switched on for 1 hour to reduce the air bubbles contain in the composite, as shown in Figure 3.2.7. Later, the specimen was left to dry at room temperature and cure for 24 hours. The similar process was adopted for the preparation of particulate reinforced SCB composites, RHA series composites and hybrid series composites.

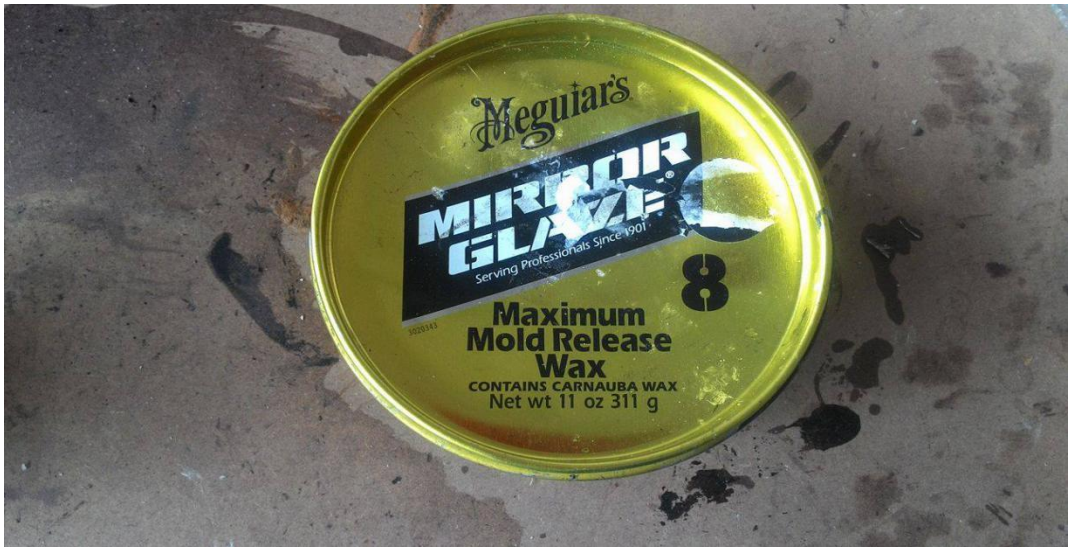


Figure 3.2.6 Mold release wax was applied on the surfaces of the tensile and flexural molds

Figure 3.2.7 The composite mixture were put into the desiccator to remove the air bubbles in the composite

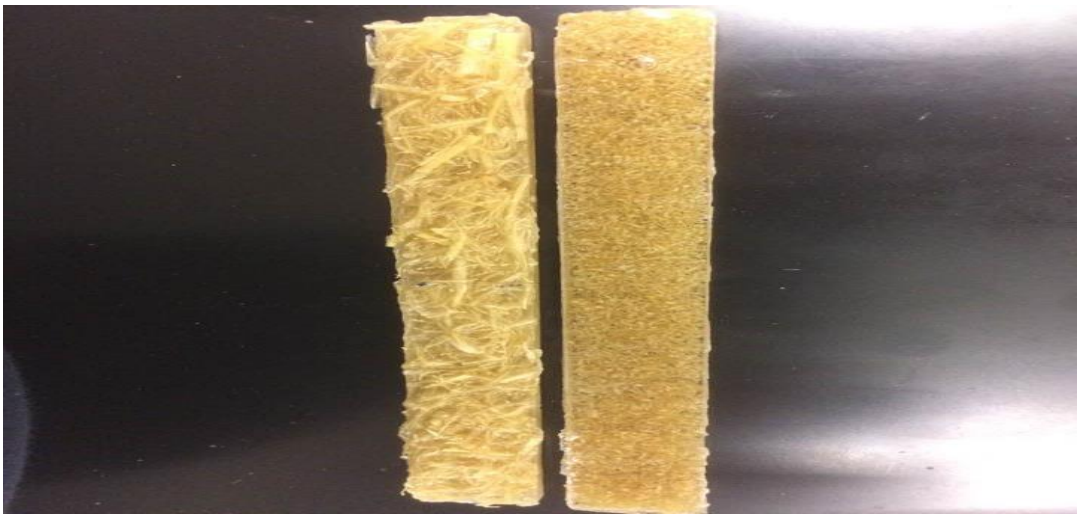


Figure 3.2.8 Composite specimen incorporated with 5% SCB fibrous and particulate form

3.3 Investigation of the mechanical properties (tensile and flexural properties) and morphology of the composite

In this section, equipment used and procedure to carry out tensile and flexural tests are described accordingly. Furthermore, equipment used and procedure of morphological studies through scanning electron microscopy (SEM) analysis are described.

3.3.1 Tensile tests

Specimens are tested using LLOYD LR 1 0K Plus Universal Testing Machine as shown in Figure 3.3.1. The test specimens are fabricated with the utilization of aluminum molds that have the standard Type 1 dumbbell shape and dimensions according to the ASTM standard D638-10. A digital micrometer was used to obtain the measurements which is width and thickness of the specimens. The standard shape of the specimen is in the form of dumbbell shape, having a dimension of $165\ mm \times 19\ mm \times 4\ mm$. A centerline was traced on the composite specimen. Specimens for tensile testing were evaluated using a 10 kN load cell and a crosshead rate of 2 mm/min with the tensile strength, tensile modulus at failure being evaluated.

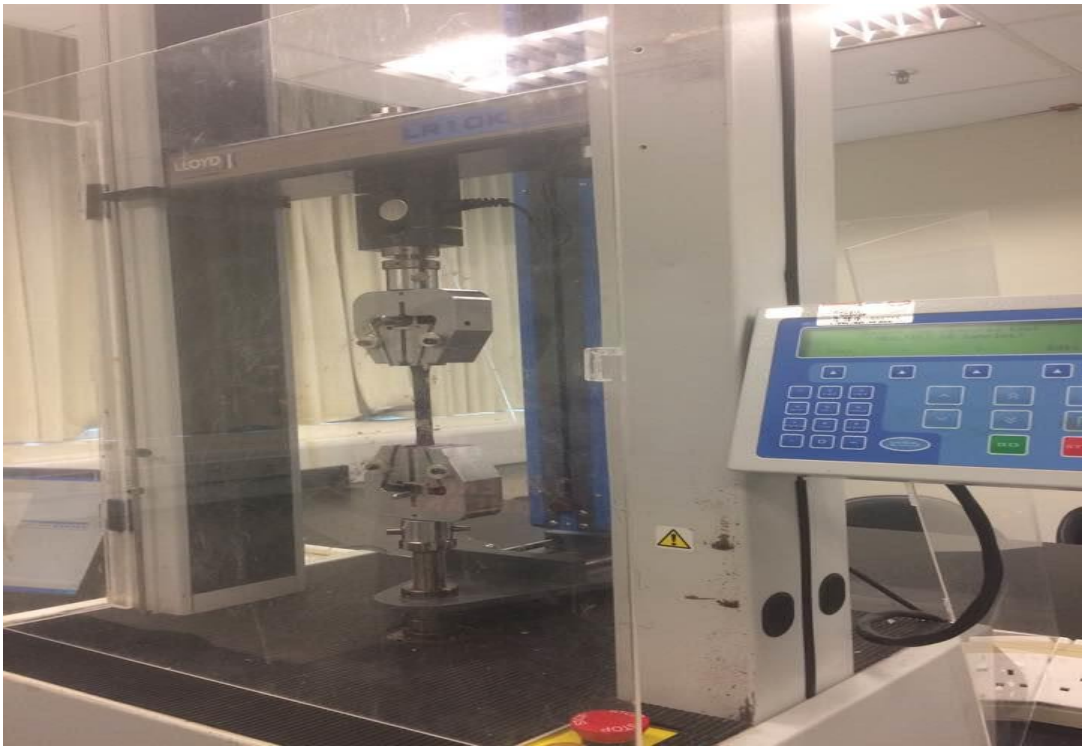


Figure 3.3.1 Tensile Test Setup for the composite specimens

3.3.2 Flexural tests

Specimens are tested using LLOYD LR 1 0K Plus Universal Testing Machine by conducting three points bending flexural tests as shown in Figure 3.3.2. The standard shape of the specimen is having a dimension of according to the ASTM standard D790-10, which is in the form of rectangular bar shape. A digital micrometer was used to obtain the measurements of the composite specimens. A centerline was marked on the composite specimen prior to the testing. The marked centerline is important as it will be the line of contact between the loading nose and the composite specimen. The flexural properties of composites were measured with a 500 N load cell and crosshead speed of 2 mm/min. The specimens were tested with a nominal span to depth ratio of 20 with the flexural strength, σ_f, flexural modulus, E_f being calculated from the following equations:

$$\sigma_f = \frac{3PL}{2bd}\left[1 + 6\left(\frac{D}{L}\right)^2 - 4\left(\frac{d}{L}\right)\left(\frac{D}{L}\right)\right] \quad (3.1)$$

$$E_f = \frac{L^3 m}{4bd^3} \quad (3.2)$$

P is the load, L is the span, b is the specimen width, d is the specimen depth, D is the deflection of the specimen at its center and m is the slope of the initial linear portion of the force-displacement curve. Five specimens for each composite composition were tested for the tensile and flexural configurations with the mean and standard deviations being presented in this work.

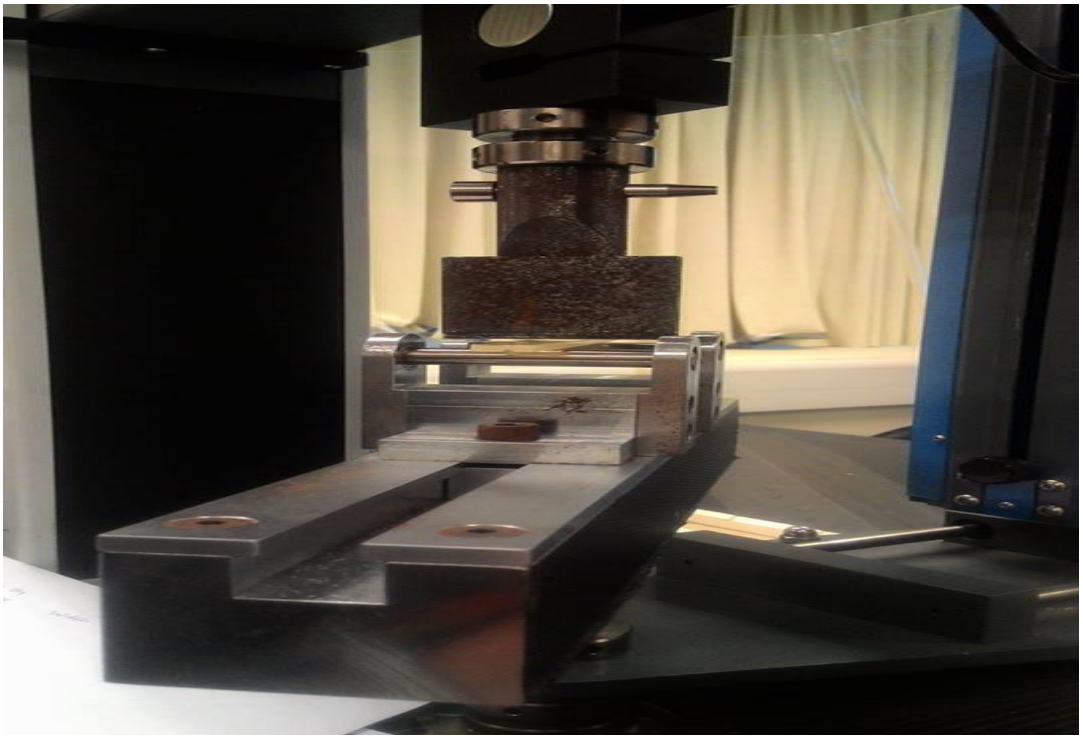


Figure 3.3.2 Flexural Test Setup for the composite specimens.

3.3.3 Scanning electron microscope (SEM)

Microstructure analysis of SCB series and RHA series composites are performed by observing the fracture surface of the composite by using scanning electron microscope (SEM). Carl Zeiss EVO 50 scanning electron microscope was utilized for the microstructural analysis using High Vacuum (HV) mode of non-conductive specimens (SE1 Detector) while the sputter coater with platinum target was applied as coating. SEM provides information on surface morphology, structure and topology of the specimens. Furthermore, SEM provides qualitative observation on the dispersion behavior of the fibers and particulates in the resin at the fracture surface of the composite. Particularly SEM can provide information on fiber dispersion or the degree of agglomeration and fiber entanglement. It can also detect the presence of micro-voids in the composite samples as well as fiber pull out from the matrix.

Figure 3.3.3 SEM for the microstructure of composite specimens

CHAPTER 4
RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter discusses the results obtained from the mechanical performance and microstructure analysis of Sugarcane Bagasse (SCB) fiber, Rice Husk Ash (RHA) and Nano Silica particulate reinforced composites. Tensile and flexural testing were conducted for all SCB series, RHA series, and Hybrid series. The mechanical properties that were analyzed in this chapter includes tensile strength, Young's modulus, flexural strength and flexural modulus. Furthermore, microstructure analysis of the composites is obtained by utilizing the scanning electron microscope (SEM) to capture the micrograph images of the fractured surfaces.

Composites that were analyzed are consist of

a) weight percentages (1%, 2.5%,5%, 7.5%) of SCB fibrous and SCB particulate in epoxy matrix under SCB series,
b) weight percentages (1%, 2.5% 5% 7.5%) of RHA particulates in epoxy matrix under RHA series, and
c) various hybrid combinations of SCB fibers, RHA particulates and nanosilica in epoxy matrix under the hybrid series.

4.2 Mechanical properties for sugarcane bagasse (SCB) series composite

4.2.1 Mechanical properties for SCB short fiber series

There are two forms of SCB fibers used as reinforcing fillers, the fibrous and particulate form of SCB fibers. In this section, the mechanical properties of SCB Short Fibers composite series are discussed. The weight percentage of SCB fillers used are 1%, 2.5%, 5% and 7.5%. The designation for SCB Series is shown in Table 4.2.1.

Table 4.2.1 Designation for sugarcane bagasse (SCB) series

No	Type of form (Fiber / Particulate)	Designation in Figure	Filler weight percentages (%)
1	SCB Fiber	SCB 1%	1
2	SCB Particulate	SCB 1%	1
3	SCB Fiber	SCB 2.5%	2.5
4	SCB Particulate	SCB 2.5%	2.5
5	SCB Fiber	SCB 5%	5
6	SCB Particulate	SCB 5%	5
7	SCB Fiber	SCB 7.5%	7.5
8	SCB Particulate	SCB 7.5%	7.5

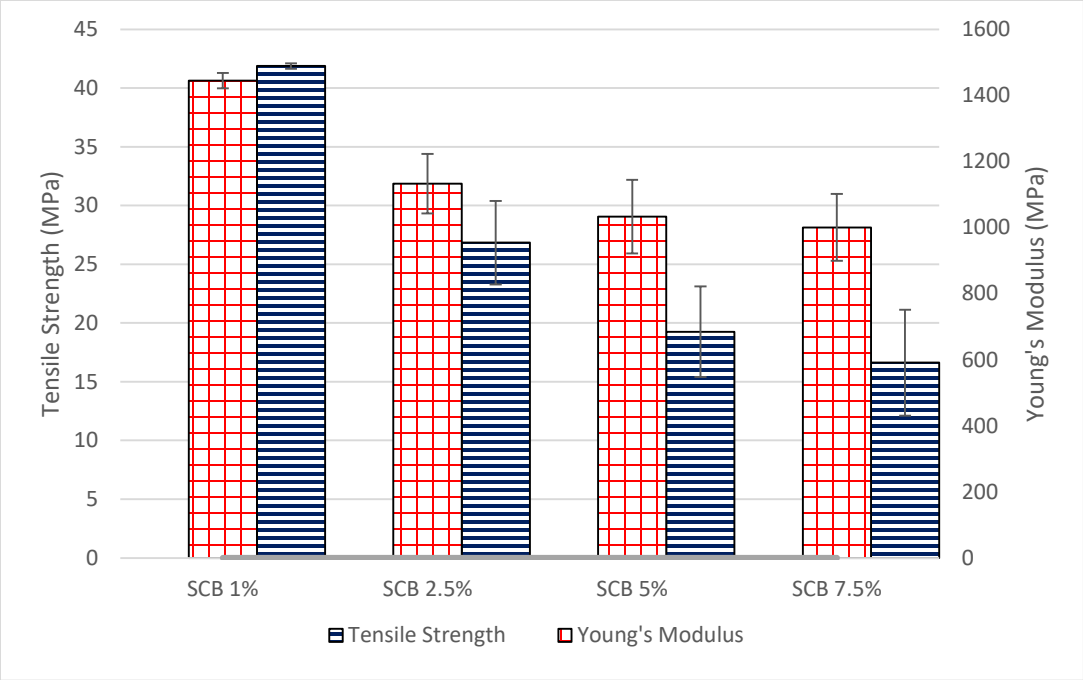


Figure 4.2.1 Tensile properties of short fiber SCB series composite

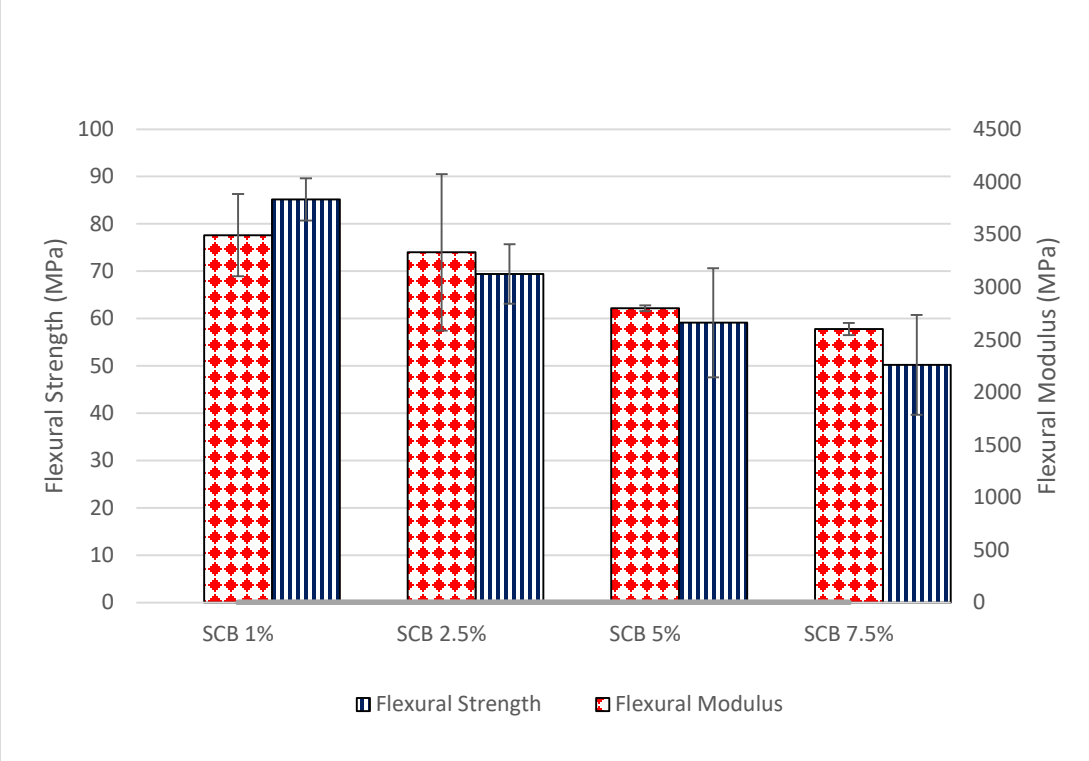


Figure 4.2.2 Flexural properties of short fiber SCB series composite

Figure 4.2.1 shows the tensile properties (Young's modulus and Tensile strength) of short fiber SCB series. SCB short fiber reinforced composites had shown to decrease gradually from the lowest fiber weightage (1%) to the highest fiber weightage (7.5%) in tensile strength. It was observed that 1wt% of SCB short fiber composite has attained the highest tensile strength and modulus which are 41.9 MPa and 1444.5MPa respectively. As for 7.5wt% of SCB short fiber composite, the tensile strength is found to be 16.6 MPa while the tensile modulus is 1000.5 MPa. Flexural properties (Flexural strength and Flexural modulus) of the short fiber sugarcane bagasse (SCB) series are shown in Figure 4.2.2. It was observed that 1wt% of SCB short fiber composite possessed the highest flexural strength and modulus which are 85.1 MPa and 3492.8 MPa respectively. As the weight percentage of SCB increased in the composite, the flexural properties gradually decreased till 7.5 wt% SCB short fiber composite with the lowest flexural strength of 50.2 MPa and flexural modulus of 2599.9 MPa. It can be observed that as the fiber content in the sample increased the tensile properties decreased. The results shown is similar with the investigation conducted by Oladele et al. 2014, SCB short fiber composites shown to have lower tensile properties at high fiber contents, which are beyond 10 wt% but the overall mechanical properties are higher than that of pure epoxy. The main reason for the reduction of tensile properties is that the bagasse fibers were not wetted properly by the epoxy resin and the fibers may have been in contact with one another. When the fibers are in contact with one another, it will hinder the proper wetting of the fibers by the matrix. The bonding between the bagasse fiber and the epoxy matrix will therefore be affected. The functional effectiveness of epoxy matrix to act as the binder material to hold fibers in position while transferring external loads to internal reinforcement was thus decreased. Hence, this will eventually lead to the reduction of the strength of the composite at higher fiber content.

According to Choi & Takahashi, 2015, following are the causes for failures in fibrous composites:

a. Shear band formation at the fiber end
b. Fiber pullout

c. Void formation

As test specimens face fiber overloading condition, the applied load is transferred from the matrix to the filler fiber by shear. Shear bands are then formed at the end of the fibers which lead to shear cracking. Shear bands grow with the increase of the load in the matrix side along the fiber matrix interface. At this stage shear cracks from the fiber end start to propagate and join together. Also due to high level of load, fibers start to break leading to tensile micro cracking in the matrix and form shear banding around the broken ends. The propagation of all of these cracks stimulates the starting of fiber pull out creating voids in the matrix. These voids grow and join together which ultimately causes the final failures of the test specimens. With higher weight percentage of fibers in the composite samples, the probability of this kind of failures increases. Therefore, lower strength and modulus are observed in higher fiber content composites. The voids for the fiber pull out are evident from the SEM image presented in Figure 4.2.3

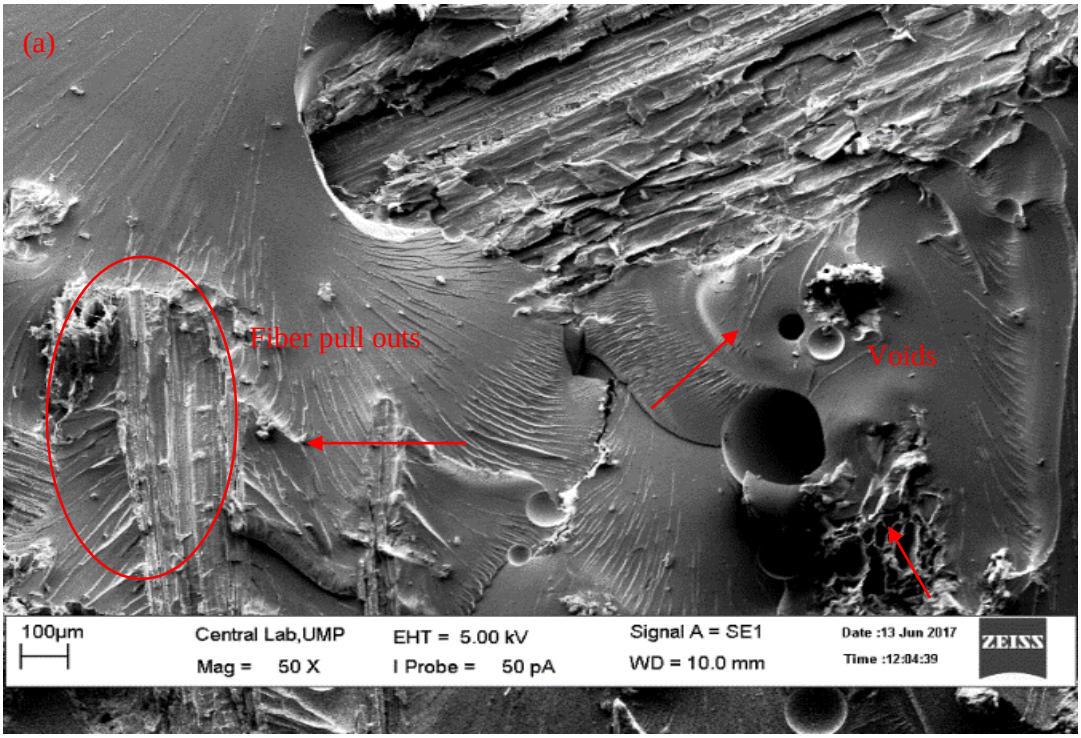


Figure 4.2.3 SEM micrograph of 7.5wt% fibrous SCB for 50 magnification after tensile fracture

According to Ramesh et.al (2013), the failure morphology of sisal-jute fibers composite was done by SEM analysis in which both fiber pull outs and fracture were also noticed. It has affected the mechanical properties of the composite produced to be less favorable. Furthermore, through the SEM micrograph that is shown in Figure 4.2.4 and Figure 4.2.5 less pull outs and voids had occurred. This is because in the case of 1 wt% of SCB short fiber composite, there were less percentage of fiber in the composite, which lead to less stress concentration at the fiber ends. Hence, there were less discontinuity and less cracks in the composite.

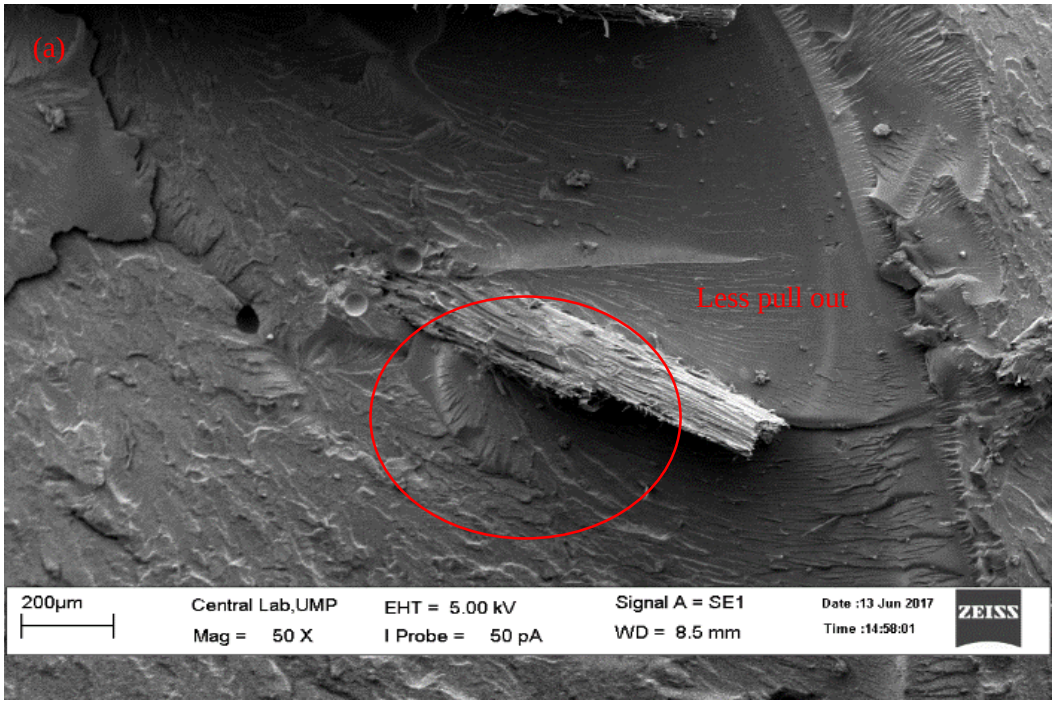


Figure 4.2.4 SEM micrograph of 1wt% fibrous SCB for 50 magnification after tensile fracture

The results shown for the case of 1wt% of SCB short fiber composite, is also in line with the investigation conducted by Oladele et al. 2014. SCB short fiber composites shown to have higher tensile properties at low fiber content, as the bagasse fibers were wetted appropriately by the epoxy resin.

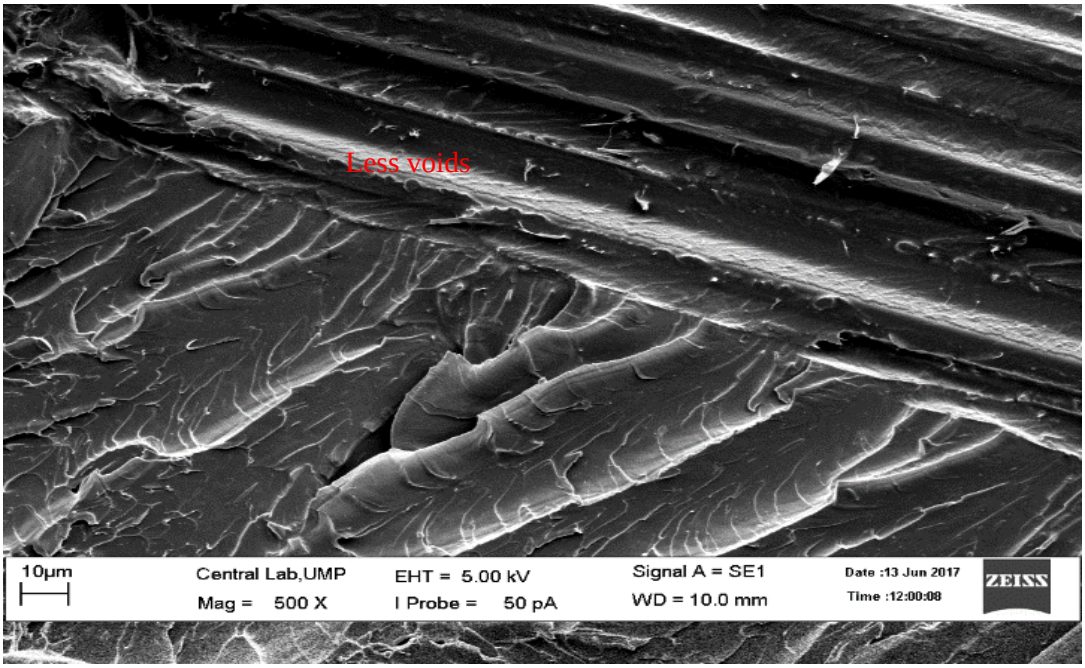


Figure 4.2.5 SEM micrograph of 1wt% fibrous SCB for 500 magnification after tensile fracture

4.2.2 Mechanical properties for SCB particulates series

In this section, the mechanical properties of composite from SCB particulate series are discussed.

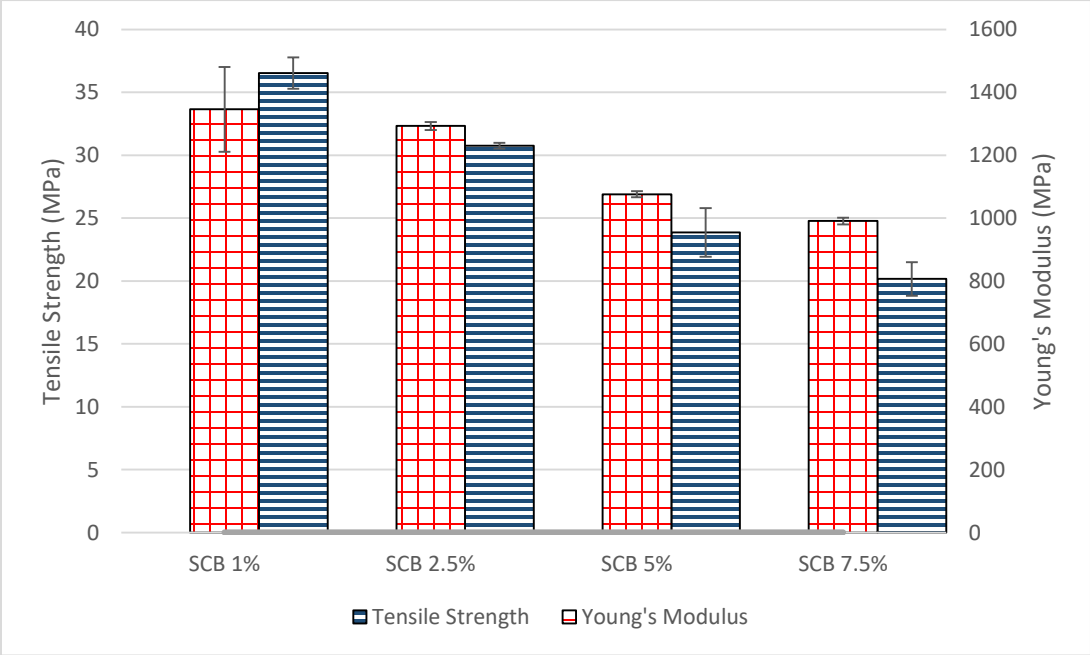


Figure 4.2.6 Tensile properties of particulate SCB series composite

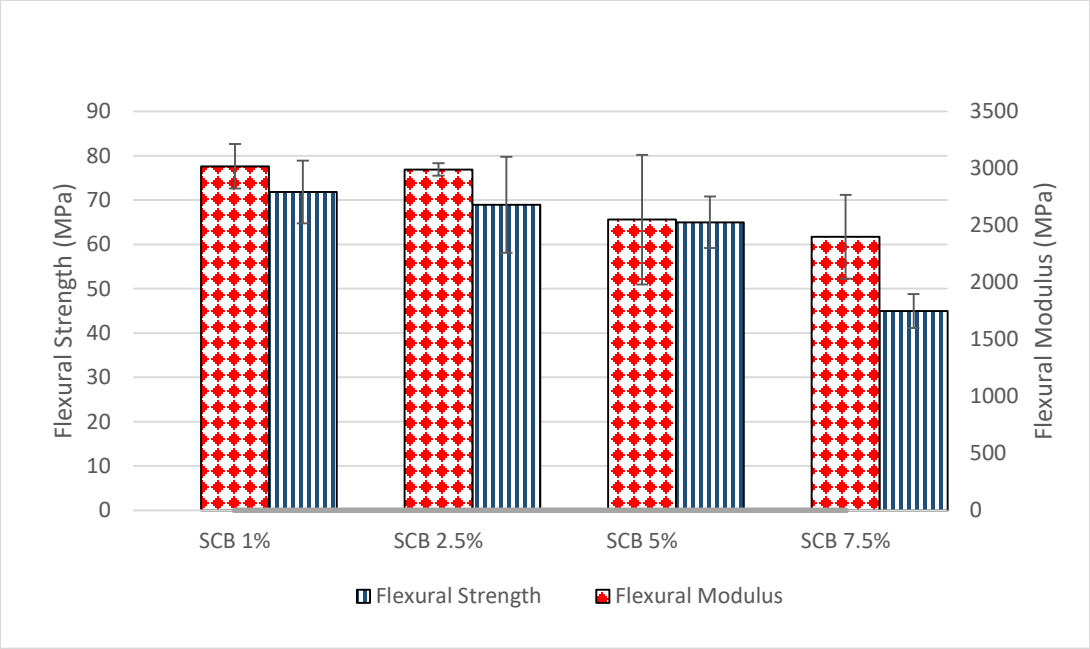


Figure 4.2.7 Flexural properties of particulate SCB series composite

The tensile properties (strength and modulus) of the synthesized specimens that have been reinforced with 1wt%, 2.5wt%, 5wt% and 7.5wt% of SCB fibers in particulate form are illustrated in Figure 4.2.6 above. In this figure, SCB particulate reinforced composites had shown to decrease gradually from the lowest fiber weightage (1%) to the highest fiber weightage (7.5%) in tensile strength. It is found that the highest tensile strength and tensile modulus of 36.5 MPa and 1345.8 MPa were obtained by 1wt% SCB particulates composite. Meanwhile the lowest tensile properties were obtained from 7.5wt% SCB particulates composite which were tensile strength of 22.8 MPa and tensile modulus of 990.6 MPa. The results shown is similar with the investigation conducted by Saini. et al. (2010), in which tensile strength of the SCB particulate filled composites also decreases with the increasing of SCB particulate weightage. The possible reasons proposed for this kind of behavior are highly possibly due to the poor interfacial adhesion between the matrix and particulates. Figure 4.2.7 presents the Flexural properties (Flexural modulus and Flexural strength) of the particulate sugarcane bagasse (SCB) series. It was observed that 1wt% of SCB particulate possessed the highest flexural strength and modulus which are 71.9 MPa and 3019 MPa respectively. The graph shown in the figure is

going on a downward trend. It was observed that 2.5wt% were still comparable to 1wt%, however as the weight percentage of fibrous particulate increased in the composite, the flexural properties deteriorated even further. The lowest flexural properties were possessed by SCB particulate 7.5 wt% composites which are 45 MPa and 2400.4 MPa for flexural strength and flexural modulus respectively.

The trend of the tensile properties (strength and modulus) and flexural properties (strength and modulus) were observed to be similar with the fibrous SCB composites which were also gradually decreasing. Although the trend in results for particulate composites are similar to the fiber composites, the mechanism of failure in particulate composites is totally different. The failure mechanism in particulate composites depends on following factors (Dong and Wu 1996):

a. Shape of the particle
b. Particle – matrix interface
c. Agglomeration

When load is applied to the composite it should transfer from the matrix to the fillers. Irregular shapes of the particle do not let this transfer happen efficiently due to stress concentrations around them. To avoid it particles are chemically treated. In this study SCB particles are treated with 2% NaOH solutions so that the interface between the particles and matrix become strong. Therefore, it is quite obvious that the failure in particulate composites start in the matrix. The beginning of matrix failure is in the polar region which is in the same direction with the applied load. The interfacial debonding in the particle matrix interface and cracks start to appear in the polar micro areas as the applied loads increase. These pave the way for voids appearing in the polar regions. When the load reaches the macroscopic failure strength some large deformations occur in the matrix between the particles causing it to lose the load carrying capacity. This mechanism prevails if the particles are evenly dispersed. However, the possibility of agglomeration makes the situation worse as

shown in Figure 4.2.8. The higher fiber weightage in the composites has higher possibilities of agglomeration. According to Fuad et al. (1993), the higher degree of agglomeration yields a more brittle composite and is more susceptible to cracking. This is because agglomerations create weak particle matrix interface and raise the stress concentrations. Hence, the load required to initiate cracking of the matrix become smaller. Therefore, higher weightage filler composites show lower strength.

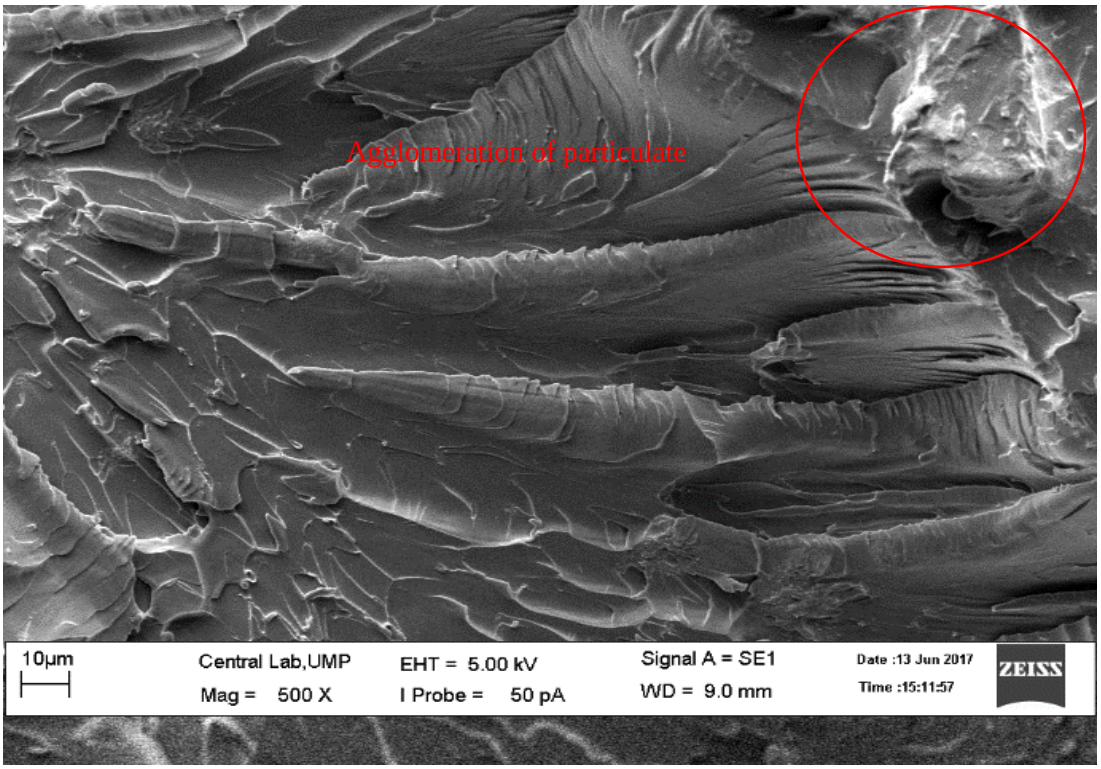
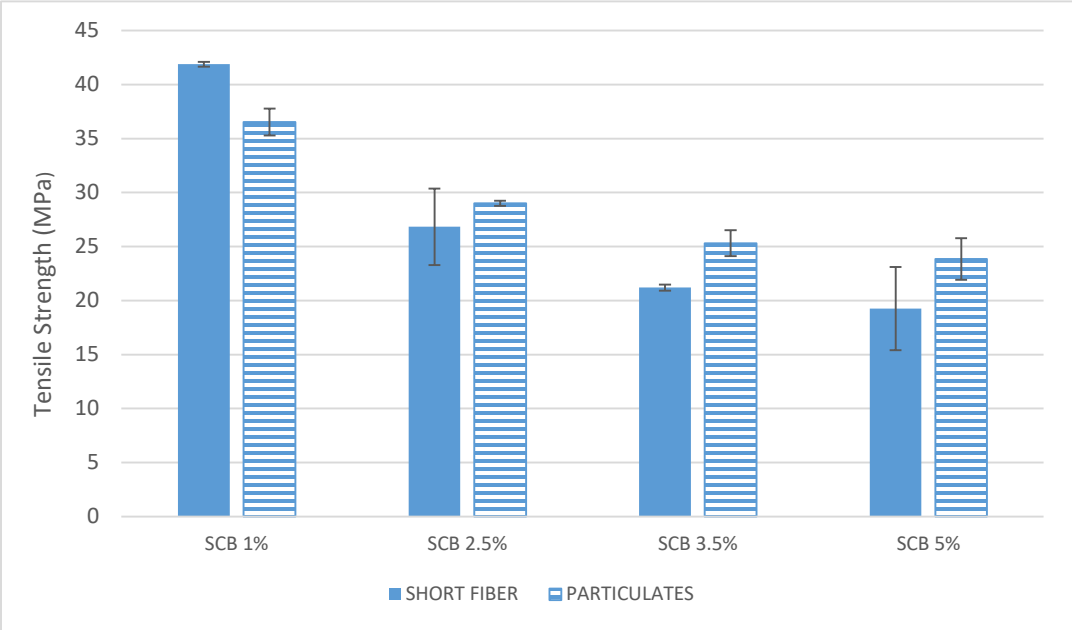


Figure 4.2.8 SEM micrograph of particulate SCB (2.5 wt%) for 500 magnification after tensile fracture

4.2.3 Comparison of SCB composite of fibrous and particulate form

In this section, the comparison between the two different forms of SCB composite, fibrous and particulate form are discussed.



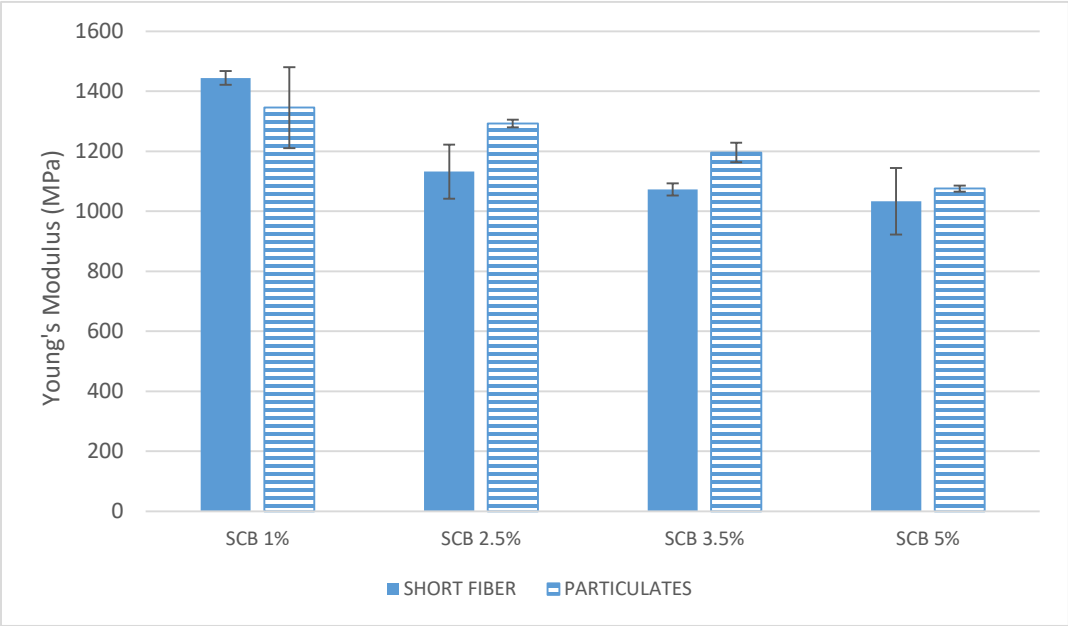


Figure 4.2.9 (a) Graph of tensile strength of SCB series composite b) Graph of Young's modulus of SCB series composite

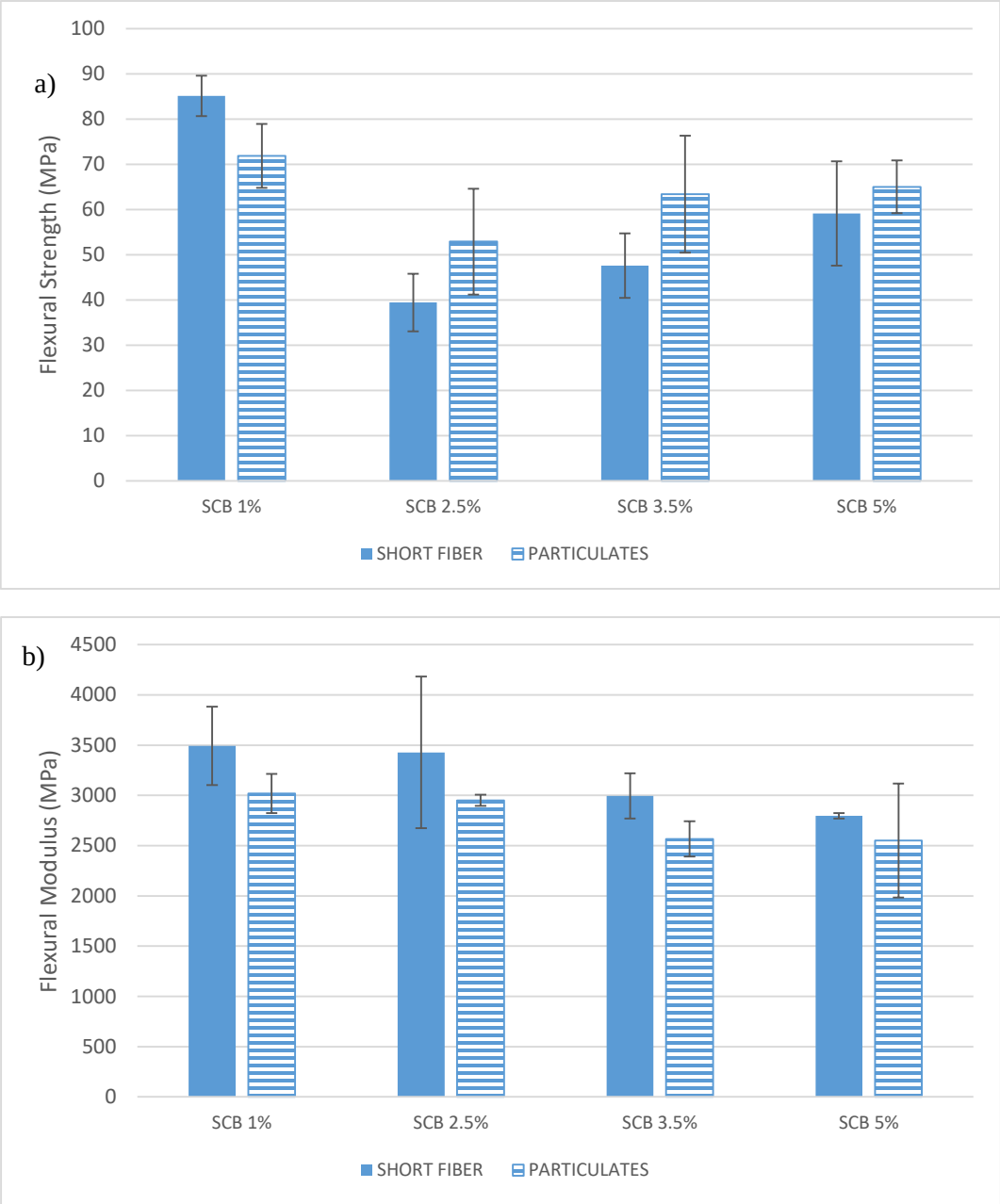


Figure 4.2.10 (a) Graph of flexural strength of SCB series composite b) Graph of flexural modulus of SCB series composite

The comparison of fibrous and particulate forms SCB composite are shown from Figure 4.2.9 and Figure 4.2.10 in terms of tensile strength, Young's Modulus, flexural strength and flexural modulus. SCB short fiber reinforced composites and SCB particulates reinforced composites are both shown to decrease gradually from the lowest fiber weightage (1%) to the highest fiber weightage (5%) in tensile

strength. The short fiber reinforced composite only showed better and improved tensile and flexural strength at lowest fiber weightage (1wt%) comparing to the pure epoxy series with no reinforcement which was 34.14 MPa and 83.03 MPa. The main reason behind this is due to more shear band formation at the fiber end, fiber pullout and void formation at higher fiber weightage. The results were in line with Rana et al. (2017), in which the tensile and flexural strengths were increased with up to 4 wt% of sisal fiber content. The addition of sisal fiber more than 4 wt% deteriorated the mechanical properties

SCB short fiber has shown overall better properties in flexural strength and modulus, which is 8.3-15.7% higher while compared to SCB fiber particulates at different weightage of fiber. It was also observed that 1wt% of SCB fibrous loading are superior in giving comparatively ideal mechanical properties while compared to specimens reinforced with SCB particulate loadings. It is more effective for the fibrous form SCB to lock into the epoxy matrix to cause improvement of the stress transfer from the matrix to the fibers. This is due to the geometrical shape and structure of the SCB in short fibrous form causing them to possess higher flexural strength.

However, the mechanical performance of the composites at higher fiber weightage favors more for SCB in particulate form. Tensile properties (tensile strength and Young's modulus) and flexural properties (flexural strength and flexural modulus) of 2.5wt%, 5wt% and 7.5wt% reinforced particulate composite is higher than that of short fiber reinforced composite. The improvement ranges from 4.1% to 14.1%. The same case also applied in flexural strength, SCB fiber particulate composites were improved from the range of 14% to 37%. This is due to the ineffective wettability of fibers in composite that has affected the bonding of fibers and matrix to be weaker. Hence, this will eventually lead to the reduction of the strength of the fibrous composite. Thus, SCB particulates possessed better mechanical properties compared to SCB fibrous form at higher fiber weightage.

4.3 Mechanical properties for rice husk ash (RHA) series composites

In this section, the mechanical properties of composite from Rice Husk Ash (RHA) series are discussed. The weight percentage of RHA fillers used are 1%, 2.5%, 5% and 7.5%. The designation for the composites for RHA series are listed in Table 4.3.1.

Table 4.3.1 Designation for RHA series

No	Designated Name	Type of filler (powder)	Filler weight percentages (%)	Total filler weight percentage (wt%)
1	RHA100	RHA	1	1
2	RHA250	RHA	2.5	2.5
3	RHA500	RHA	5	5
4	RHA750	RHA	7.5	7.5
5	NRHA100	RHA, SiO_2 (Nano Silica)	0.5,0.5	1
6	NRHA250	RHA, SiO_2 (Nano Silica)	1.5,1	2.5
7	NRHA500	RHA, SiO_2 (Nano Silica)	4,1	5
8	NRHA750	RHA, SiO_2 (Nano Silica)	6.5,1	7.5

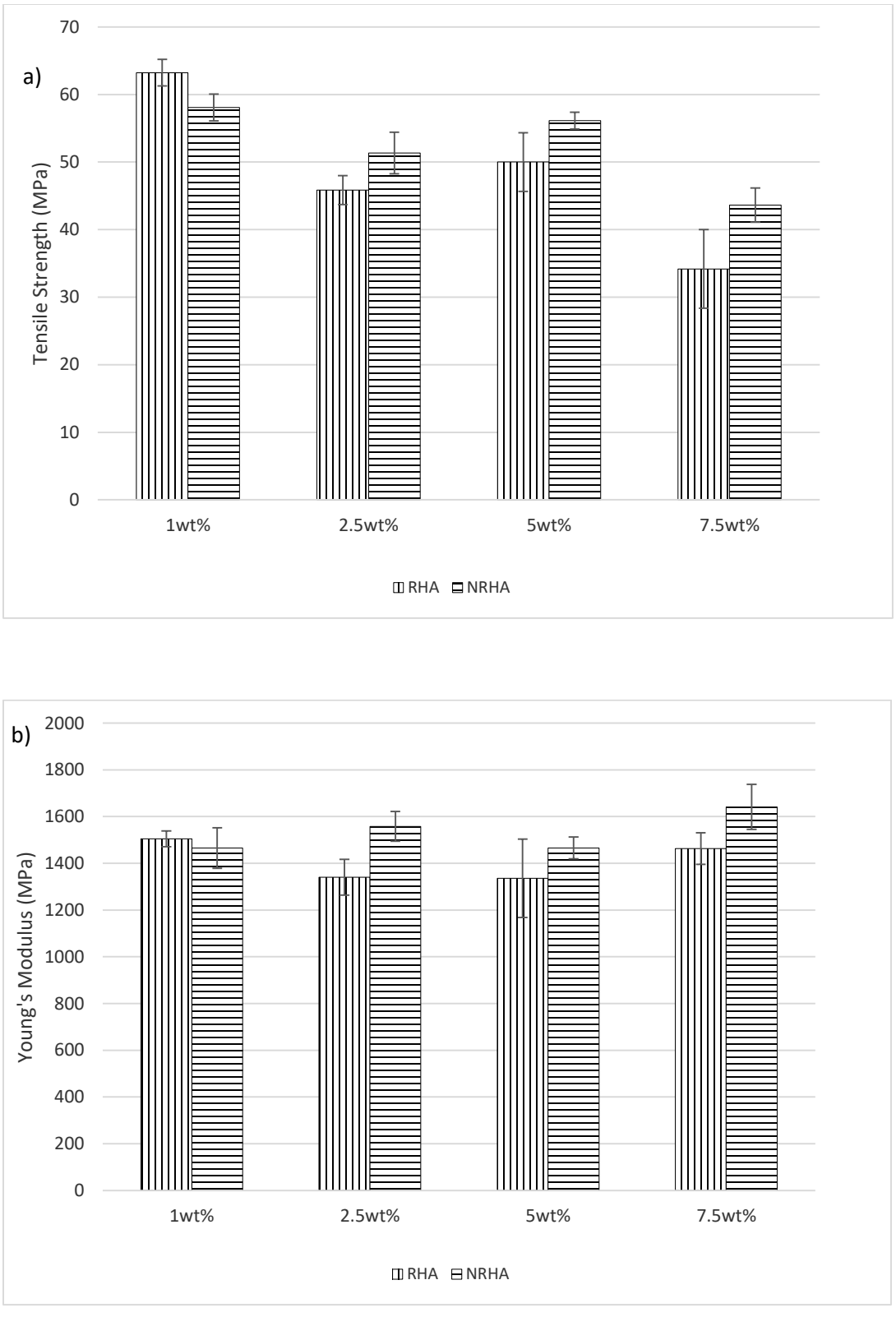


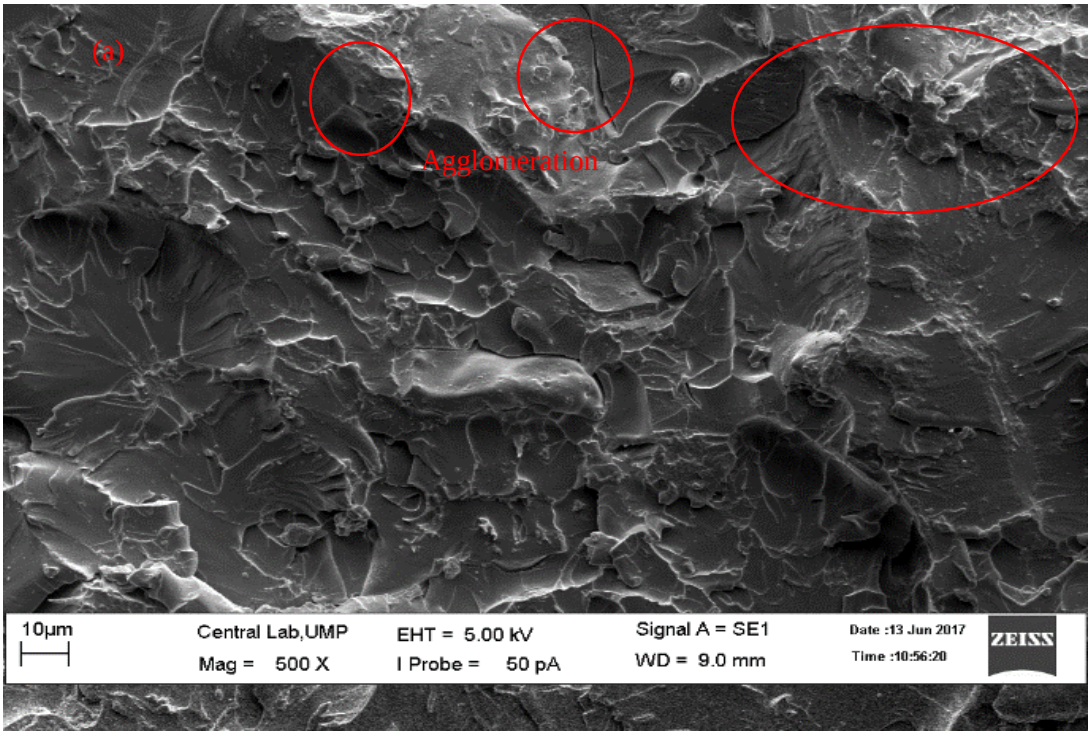
Figure 4.3.2 (a) Graph of tensile strength of RHA series with different weight percentages (b) Graph of Young's Modulus of RHA series with different weight percentages

The experimental results on tensile properties (Young's modulus and Tensile strength) of the composite that have been reinforced with 1 wt%, 2.5wt%, 5wt% and 7.5wt% of RHA Series (pure RHA and RHA with the addition of nanosilica) were illustrated in Figure 4.3.2. It was observed that RHA100 composite possessed the highest tensile strength of 63.23 MPa while NRHA750 has the highest Young's modulus of 1641.11 MPa. An important observation to be indicated was that the higher loading of RHA in the epoxy matrix contributed to the higher Young's modulus of the composite. According to Ayswarya et al. (2012) as RHA filler is capable in contributing uniform distribution of stress which in turn aiding to delay the rupture of material when subjected to exterior stress. It was also observed that the lowest tensile strength is possessed by RHA750 with tensile strength of 34.18 MPa followed by NRHA750 of 43.62 MPa. This result was in line with Fernandes et al. (2018), increasing filler loadings caused a slight increase in Young's Modulus but reduced in terms of tensile strength of the composites.

In the previous section of Chapter 4.2, the mechanism of failure in particulate composites is described and analyzed according SCB of particulate form. In this section, a similar form of filler, RHA particulate is utilized as reinforcement in the composite. RHA particulate is made up of silica, which is of similar content to nanosilica particulate which is usually used as a filler material in composite. The initial hypothesis suggested that the incorporation of RHA can fill in the gap of spaces in the matrix, thus improving the mechanical performance of the filler while the presence of nanosilica particulates will contribute to the filling of the smaller space in the matrix. The failure mechanism in particulate composites depends on following factors (Fuad et al. 1993); (Dong and Wu 1996) :

a. Shape of the particle
b. Particle – matrix interface
c. Agglomeration

Similar to SCB particulate, the irregularity of the shape of RHA particle also caused the stress transmission from the primary phase to the fillers to be inefficient due to the stress concentrations around them. When stress is applied on the material by loading, the matrix in the polar region of the particulate yields and de-bonds from matrix first, and then as the load increases, the yielding and debonding zone gradually extends. At a certain stress level, the particulate is liable to entirely or partially debond from the matrix and thus forming large voids at the polar region of the particles. The load carrying capacity of the material are affected severely at this point. All the particulate-filled material in the research have undergone the similar damage evolution process. The difference between them is that the stress level of the initial debonding which differs in each case. The main layback in this case is due to the agglomeration of particulates that occur in the composite as the RHA filler increased. The tensile strength of powder composites is known to strongly depend on the dispersion of the filler in the composite. According to Fuad et al. (1993), more agglomeration that occurred in the composite system causes a more brittle composite to be formed and is more liable to cracking. When agglomeration of particulate occurs in the composite, the stress concentrations are formed around these agglomerates, which then lead to the initial cracking of the matrix when subjected to tensile loading. This reflects in the results that were obtained as voids appeared in the polar zone of the particulates in RHA750 composites which is shown in SEM micrographs of Figure 4.3.3 grew along the tensile direction. The results are also in line with the investigation done by Kabir et.al (2016) as increased volume fraction of RHA produced a more brittle composite with increased numbers of cracks. Hence, the mechanical properties of the composite, particularly the flexural strength decreased when more RHA were induced into the composite.



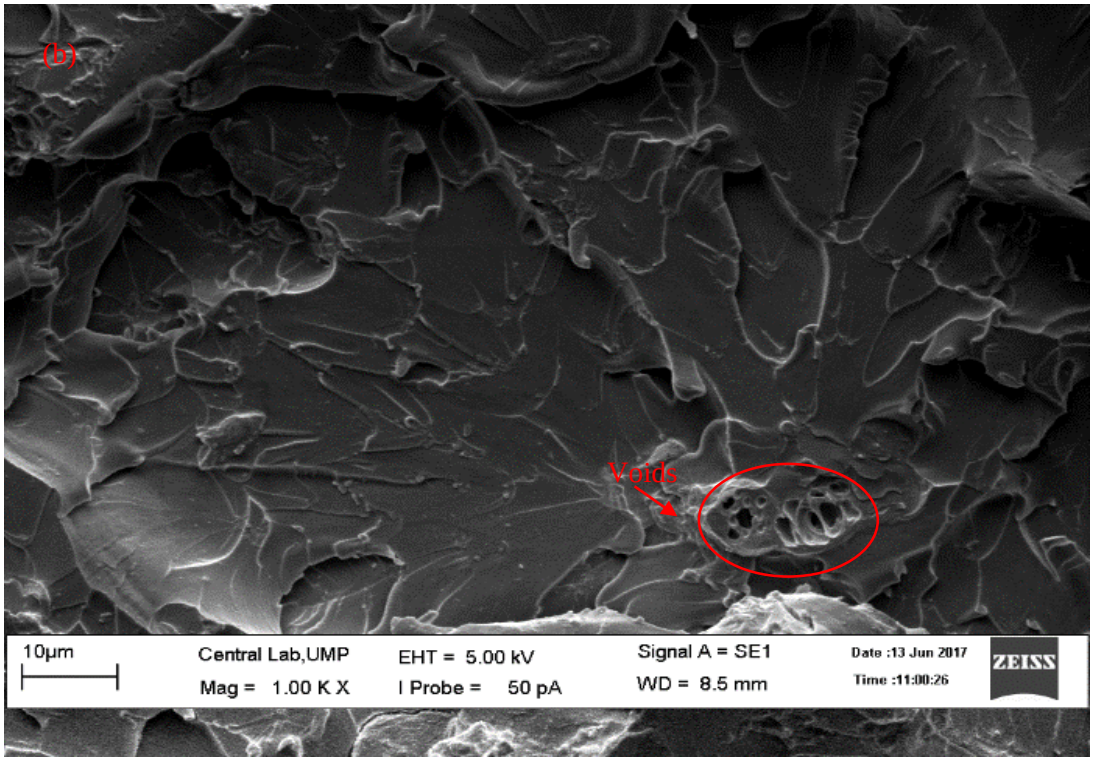
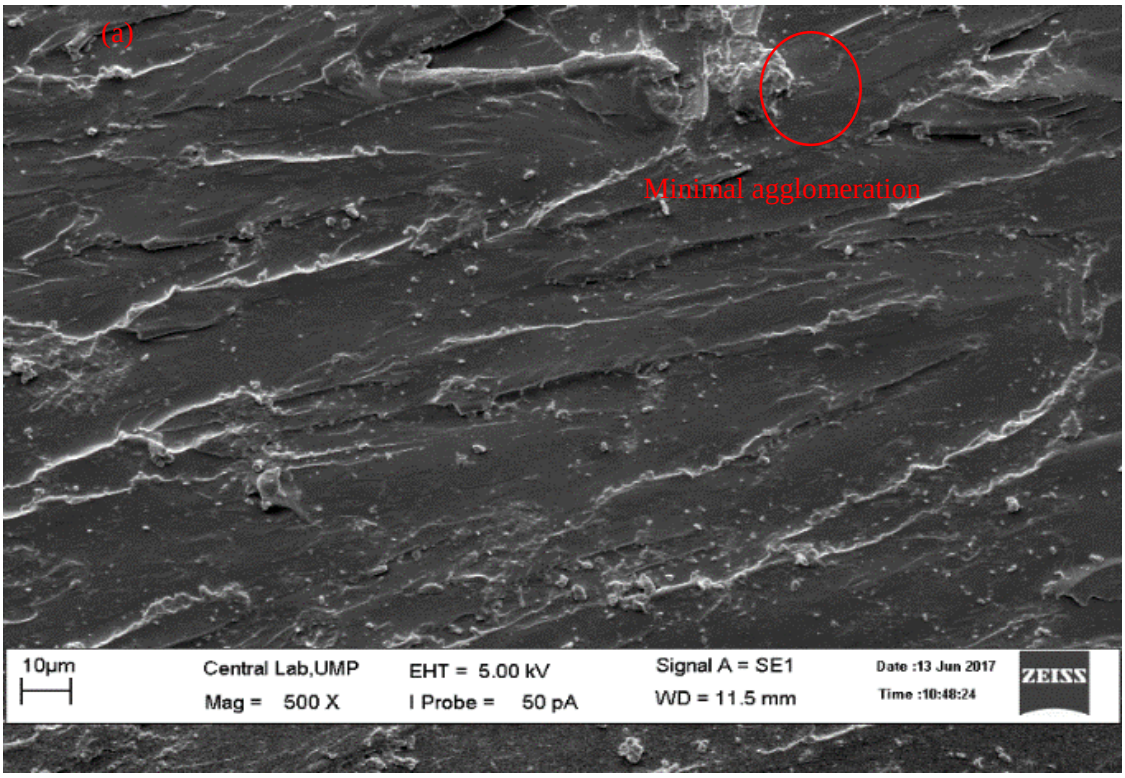


Figure 4.3.3 SEM micrograph of RHA750 (RHA 7.5 wt%) for (a) 500 magnification (b) 1000 magnification after tensile fracture

As for the results shown for the case of RHA100 composite, it is also line with the investigation conducted by Ayswarya et al. (2012). Both RHA and NRHA series composites shown to have higher tensile properties at low filler content. Through the SEM micrograph that is shown in Figure 4.3.4, less agglomerations had occurred. This is because when there were less percentage of fillers in the composite, less agglomerations are formed. Hence less stress concentration will accumulate around the agglomerates and there were less cracks in the composite.



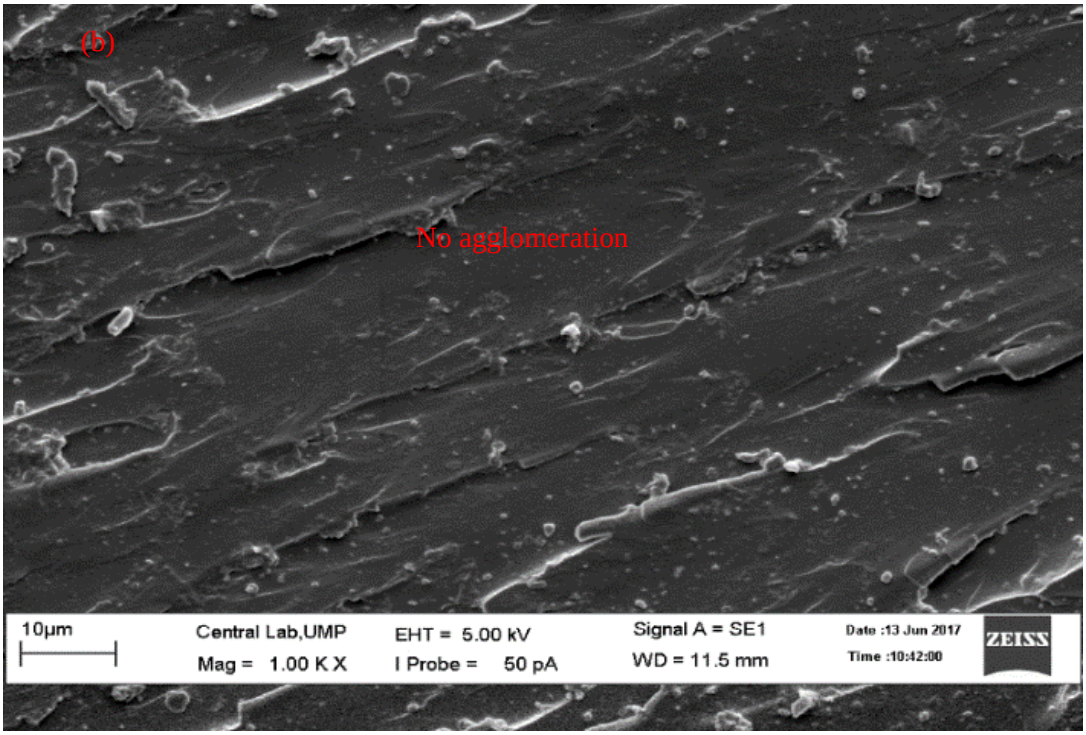


Figure 4.3.4 SEM micrograph of NRHA100 (RHA 0.5 wt%, N-SiC 0.5%) for (a) 500 magnification (b) 1000 magnification after tensile fracture

To improve the mechanical performance of the composite, the agglomeration problem should be eliminated. This confirmed that there is an optimum loading of RHA and nanosilica to give the best reinforcement for the composites, which in line with similar research carried out by Kabir et al. (2016), Ayswarya et al. (2012), Turmanova, Dimitrova, and Vlaev (2008) and Fuad et al. (1993).

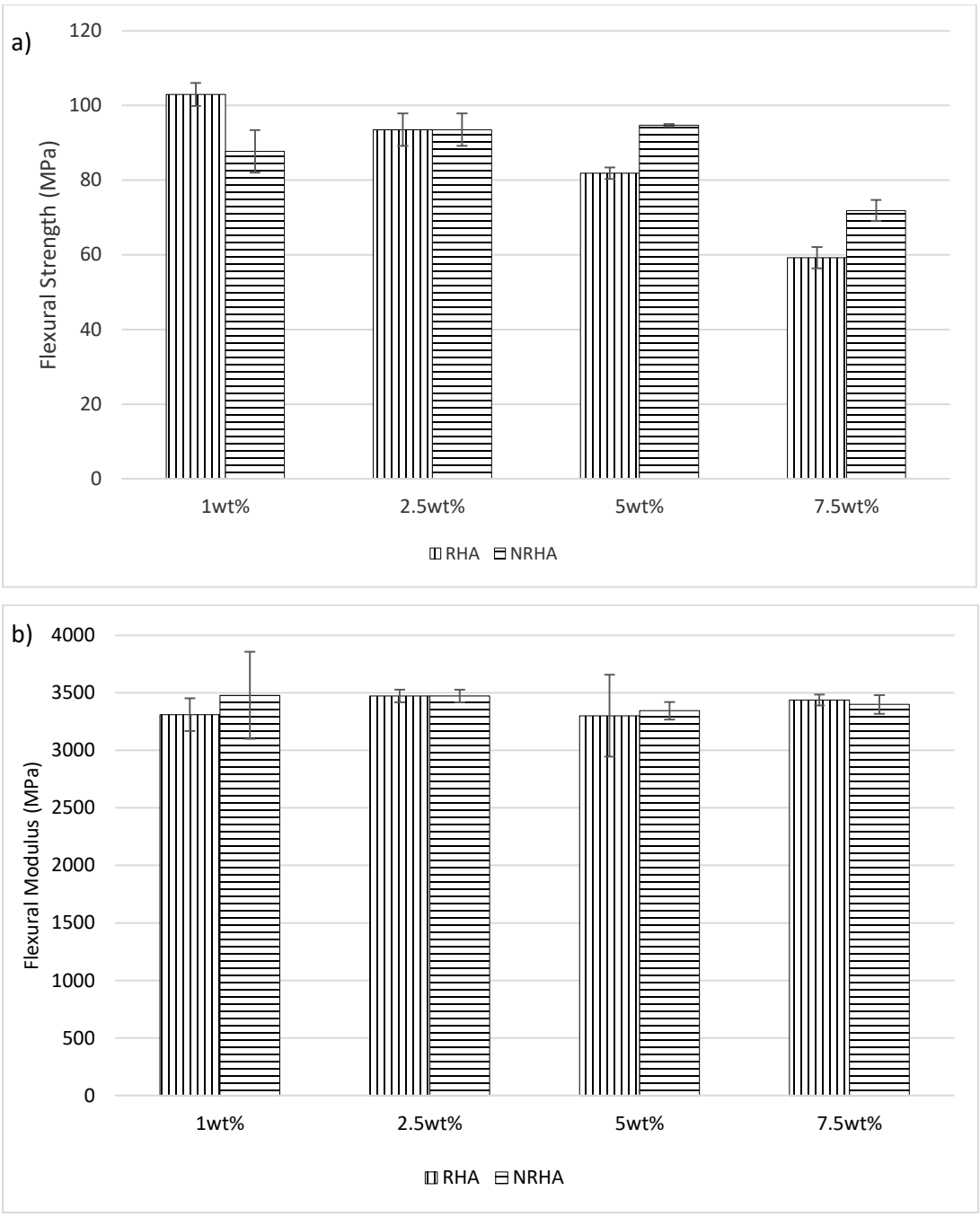


Figure 4.3.5(a) Graph of flexural strength of RHA series with different weight percentages (b) Graph of flexural modulus of RHA series with different weight percentages

Flexural properties (flexural strength and flexural modulus) are shown in Figure 4.3.5. It was noticed that the trends were almost in line with that of tensile properties testing results for the same set of specimens. RHA100 specimens showed the highest flexural strength, which is 103 MPa. It is also worth to take note that the

flexural modulus of the sole-RHA series specimens showed relatively high values, with the highest value possessed by RHA250, with 3472 MPa. An observation to be indicated was that with the addition of nanosilica, the flexural modulus improved comparing to RHA100. According to Fuad et al. (1993), the higher degree of agglomeration yields a more brittle composite and is more likely to initiate cracking of the composite. As the dispersion of RHA within matrix would be different for every single specimen, the possible agglomerations and void spaces formation also lead to the different strains experienced which in turn led to result fluctuations.

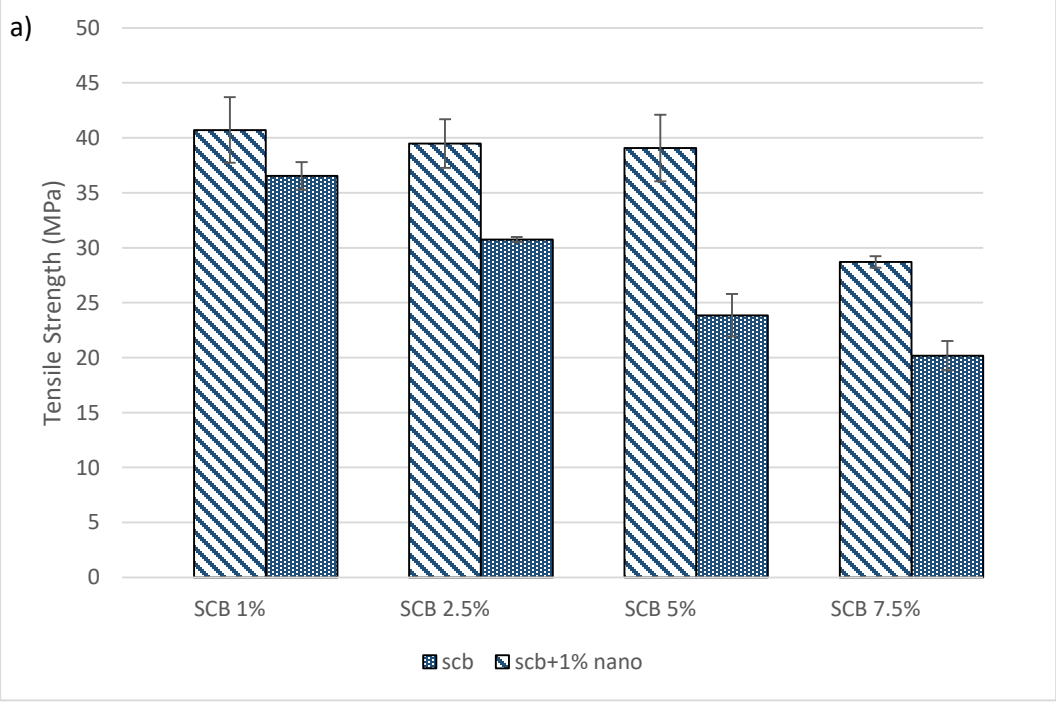
Through this study, it was revealed that addition of nano silica and RHA did improve the mechanical properties of the composite in RHA series. The tensile strength of RHA 100 improved by the most, which is 46.1% compared to pure epoxy while RHA 750 were also comparable to the pure epoxy. Besides that, the dispersion of RHA is different in different percentages as the possible agglomerations and void spaces formation also lead to different strains experienced and hence affecting tensile properties of the system, which in turn led to result fluctuations. RHA in this case had contributed for the uniform distribution of stress which in turn aiding to delay the rupture of material when subjected to exterior stress.

4.4 Mechanical properties of the hybrid series composite

In this section, the mechanical properties of hybrid composite with different reinforcement combinations were analyzed. Theoretically, it was anticipated that when SCB, RHA and nanosilica are used in combination, the well dispersed RHA can fill up the micro-voids and nanosilica filling up the nano-voids that have been formed in the epoxy matrix to grant the material with better resistance to inhibit the external stress; while SCB fibers aid in the transferring and bearing the undesired loads.

4.4.1 Hybrid sugarcane bagasse (SCB) series

In this section, SCB fiber particulates composites from Section 4.2.2 were compared with hybrid SCB fiber particulate composites with the addition of nanosilica particulate. The mixing method for both sets of composites are by utilizing the ultrasonic processor.



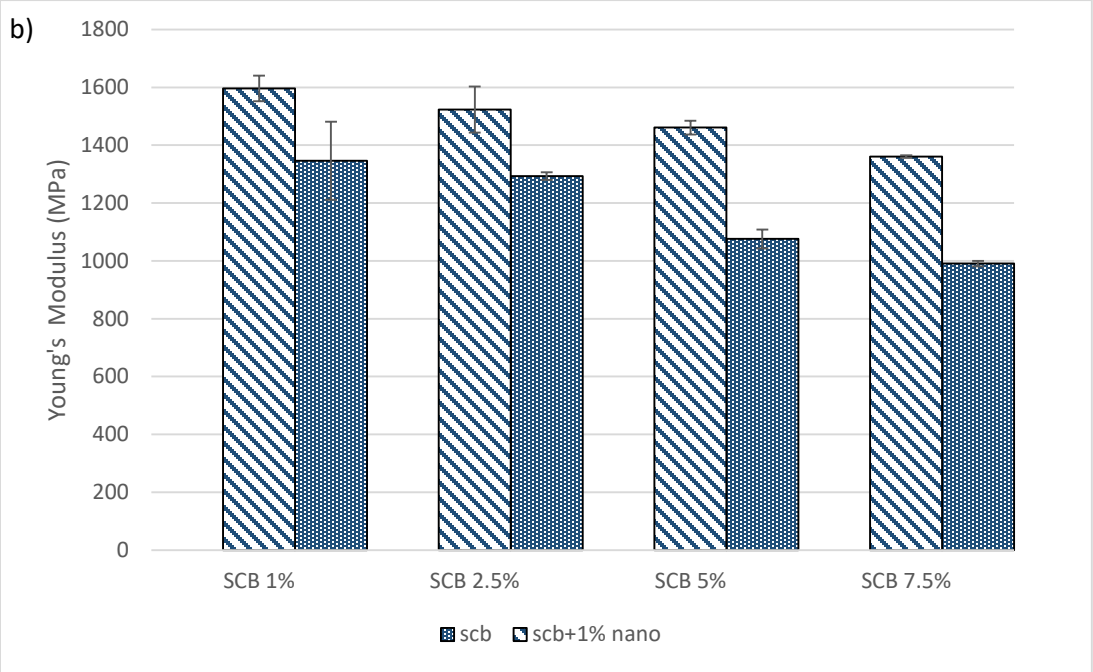


Figure 4.4.1 Graph of comparison between SCB composites and SCB composite with addition of nano-silica in terms of (a) tensile strength (b) Young's modulus

Figure 4.4.1 shows the graph of comparison between SCB composites and hybrid SCB composites with addition of nano-silica in terms of tensile strength and Young's modulus. From the figure, it is shown that composite reinforced with SCB 1wt% and nanosilica possessed the highest tensile strength and modulus of 40.7 MPa and 1595.9 MPa respectively. With the addition of nano-silica particulates, it is observed that the tensile strength and tensile modulus of the SCB composites were successfully improved. The improvement ranges from 11% to 63% for tensile strength and 18% to 37% for Young's modulus respectively compared to the pure SCB series composite. However, for composite of both series with or without the addition of nanosilica particulate, the graph trend shown in Figure 4.4.1 is a downtrend. The similar trend was also observed for both particulate and fibrous SCB series in Figure 4.2.1 and 4.3.1 of Chapter 4.2. The reason behind this is also attributed to the effects of agglomeration due to poor dispersion of the fillers within the matrix as agglomerations generate weak particle matrix interface. This phenomenon in the

composite also raise the stress concentrations. Hence, when more stress concentrations were created, the load required to initiate cracking of the matrix become smaller. Therefore, higher weightage filler composites show lower strength.

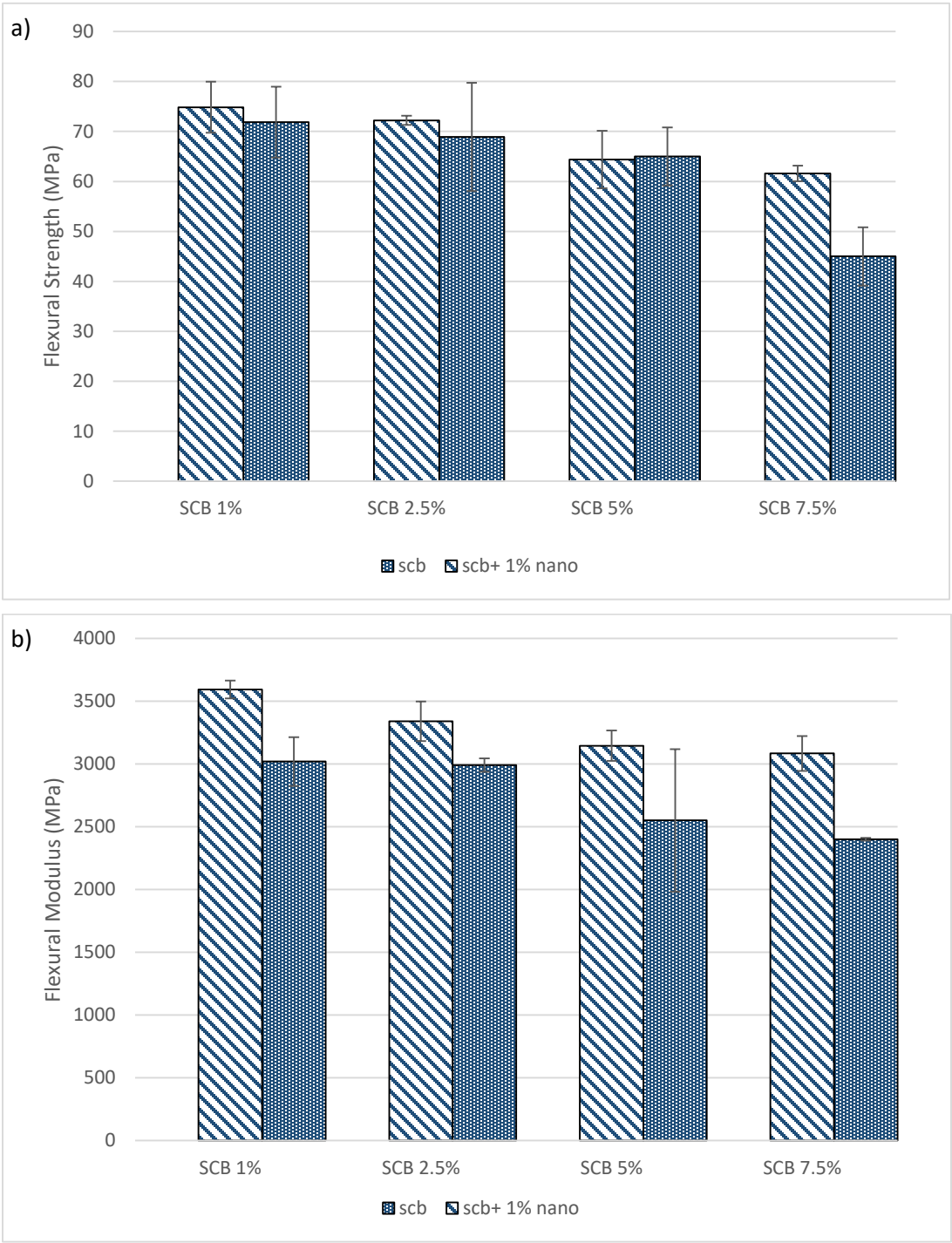


Figure 4.4.2 Graph of comparison between SCB composites and hybrid SCB composite with addition of nano-silica in terms of (a) flexural strength (b) flexural modulus

Figure 4.4.2 shows the graph of comparison between SCB composites and hybrid SCB composite with addition of nano-silica in terms of flexural strength and flexural modulus. The highest flexural properties were possessed by composite reinforced with 1wt% SCB and nanosilica having flexural strength and modulus of 74.8 MPa and 3592.7 MPa respectively. The results obtained was in line with the research done by Dittenber et al. (2012), which indicated that the addition of nano-silica particulate is able to increase stiffness at 10% as the particulates fill in the voids in the composites. It was noticed that the trends were in line with the tensile properties results except for SCB 5wt% that might be due to distinctive dispersion of fibers in the particular specimens that affected the tensile properties of the system, which in turn led to result fluctuations. The improvement ranged from 11% to 28% for flexural strength and 4% to 36% for flexural modulus.

From the results, it can be concluded that the nano-silica particles have improved the mechanical performance for both tensile and flexural properties of the composite due to its large surface area and its smooth nonporous surface. When compared to the pure epoxy, tensile properties improved up 15% but dropped when the SCB weightage increases to 7.5wt%. From the research done by Rong et al. (2015), in the case of cementitious composites, when nano-silica particles were added up to 3 wt%, the flexural strength of the composite was found to be the highest. However, when more nano-silica particles were added to the composite system, agglomerations started to form and causing a decline in the mechanical properties.

4.4.2 Hybrid Series for RHA, SCB and nano-silica particulates reinforced composite

In this section, different weightage of SCB fiber particulates and RHA particulate were reinforced with the addition of nanosilica particulate into epoxy resin to produce hybrid composite. The designation for the hybrid series is listed in Table 4.4.2.1.

Table 4.4.2.1 Designation for hybrid series composites

Designation		Filler Percentage (wt%)			
		SCB Particulate	SCB Fiber	RHA Particulate	Nanosilica Particulate
Hybrid Fiber (HF)	HF 300	0	1	1	1
Hybrid Fiber (HF)	HF 700	0	3	3	1
Hybrid Particulate (HP)	HP 300	1	0	1	1
Hybrid Particulate (HP)	HP 700	3	0	3	1

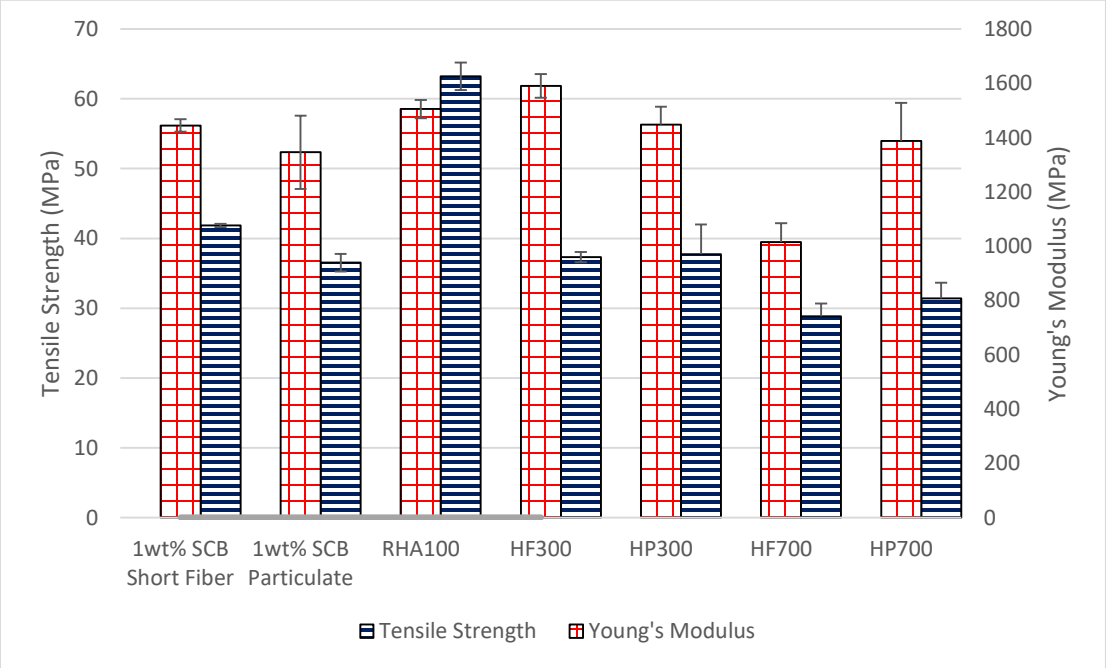


Figure 4.4.3 Comparison between best performing sole-reinforced composite and hybrid composites in terms of tensile strength and Young's modulus

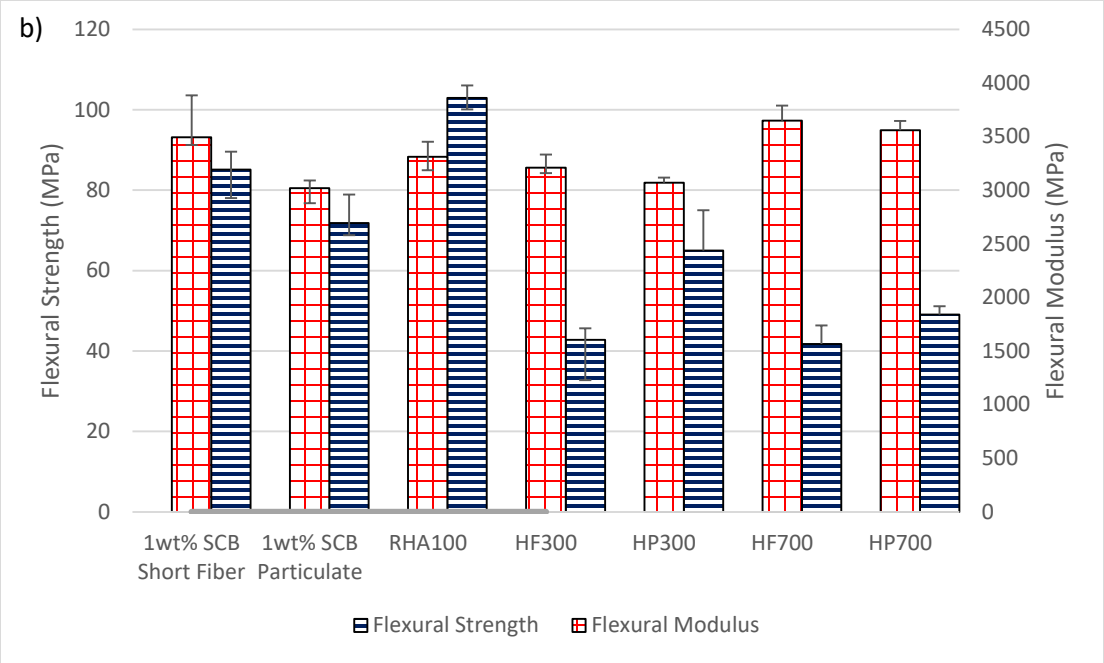


Figure 4.4.4 Comparison between best performing sole-reinforced composite and hybrid composites in terms of flexural strength and flexural modulus

The first combination, HF 300 was adding 1wt% of SCB in fibrous form together with 1wt% of RHA and 1wt% of nanosilica particulate into the epoxy resin. From Figure 4.4.3, the tensile strength of HF 300 is 37 MPa while the tensile modulus is found to be 1590.2 MPa. While tensile properties for HP 300 is 37 MPa for tensile strength and 1447.3 MPa for tensile modulus. It was found out for tensile properties, fibrous form SCB hybrid composites performed better in low percentage of filler mixtures. As for particulate form SCB hybrid composite performed better in high percentage of filler mixtures. From Figure 4.4.3, HP 300 possessed a tensile strength of 31.4 MPa while HF 700 is found to be 28.8 MPa. For Young's modulus, HP 700 with 1388.2 MPa was found to be higher than HF 700 of 1015.2 MPa. The reason behind this situation is that the excessive amount of fiber is spotted in case of higher fiber content and it had led to the entanglement and agglomeration of fillers in the composite. This had influenced the efficiency of stress transfer between fibers as wettability of the fibers were not substantial. Hence, tensile properties of the specimen with excessive SCB in fibrous form (HF 700) is much lower. However, as for flexural properties, it is worth noticing that flexural modulus of HF

700, 3647 MPa, is found to be the highest among all composites. This is due to fibers were more capable in preventing the deformation under the presence of bending loads and fatigue stresses. As suggested by Smith and Yeomans (2009), short fibers polymer composite offer modest strength and stiffness compared to other form of polymer composites.

However, all the hybrid composite specimens have lower tensile and flexural strength compared to the sole-reinforced composite (1wt% SCB fibrous, 1wt% SCB particulate, 1wt% RHA). This is due to fiber entanglements and agglomeration that has been induced among the fillers during the mixing process in the hybrid composite. d etc.). It was not only difficult to thoroughly mix the resin mixture in assuring an uniform and even dispersion of additives within the matrix, the viscosity of mixture also become higher and results in the rather challenging manufacturing process. In addition, it can also lead to catastrophic and earlier failures SEM micrograph in Figure 4.4.5 portrayed the hybrid particulate SCB composite which has obviously possessed better mechanical properties due to good bonding. While as a contrast, SEM micrograph in Figure 4.4.6 portrayed the hybrid fibrous SCB composite which showed serious fiber de-bonding due to excessive fibers entanglement. Similar results were published by Zainal et.al (2019), when the SCB filler was observed to be pulled out from the matrix and showed more detachments of SCB filler from the matrices. The SEM micrographs of fractured surface of hybrid composites observed to be having voids between the fillers and matrix. Thus, showing the poor adhesion between the filler and matrix. When the fibers added to the matrix has reached a certain amount, the fillers might have agglomerated or entangled. These agglomerations or entanglement therefore become stress concentrators, which are the cause of reduction in flexural strength (Rout and Satapathy, 2012). When excessive fibers are embedded in the composites, unnecessary interaction between the fibers will decrease the tensile strength of the composite. The results are in line with the research conducted by Fuad et al. (1993), Ayswarya et al. (2012), Rout and Satapathy (2012) and Kar et al. (2018).

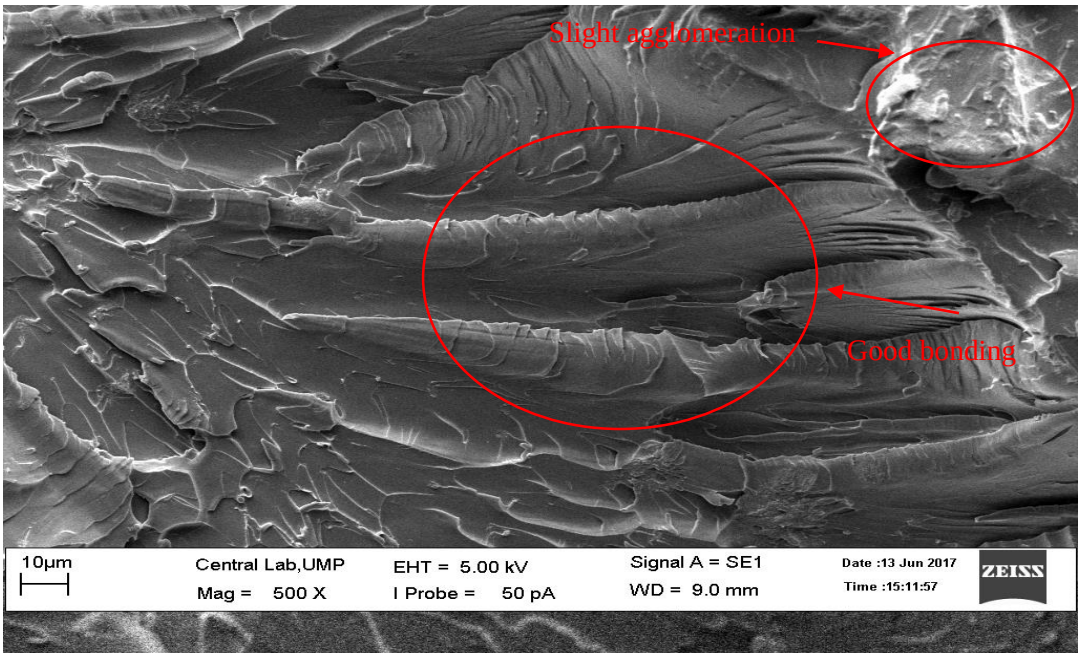


Figure 4.4.5 Hybrid SCB particulate (SCB particulate 3 wt%, RHA particulate 3 wt%, N-SiC 1%) composite with addition of nano-silica

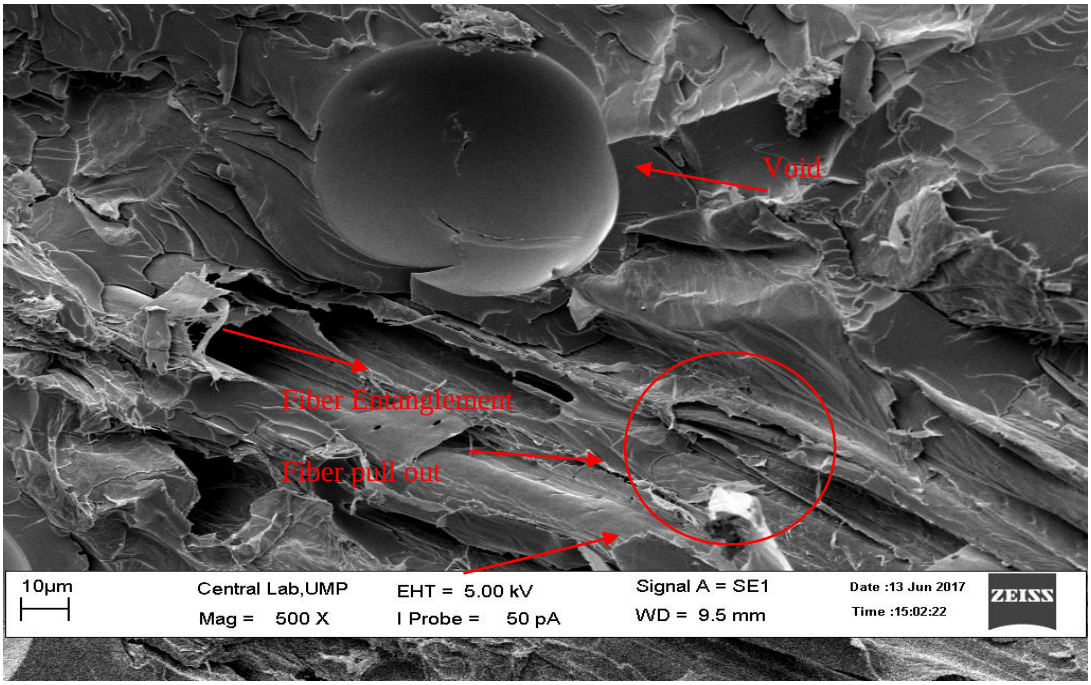


Figure 4.4.6 Hybrid SCB fibrous composite (SCB fibrous 3 wt%, RHA particulate 3 wt%, N-SiC 1%) with addition of nano-silica

When proper modification and manufacturing procedures are applied, composites reinforced with SCB showed improvement in the mechanical properties such as Young's modulus and flexural modulus. However, when the percentage of SCB was added until a certain amount, the mechanical properties of the composites were

noticed to decrease significantly. This is due to the entanglement of fibers in the resin, weak interfacial bonding between the filler and matrix in the composite and relatively rough surface finish of the composites. Hence, current study involves utilization of rice husk ash (RHA) and nanosilica as two other reinforcing fillers in the making of a hybrid composite in order to improve the mechanical properties by filling up the micro and nano-spaces in the composite system.

From the results, it is worth highlighting that all the sugarcane short fiber reinforced epoxy composites obtained higher flexural modulus compared to sugarcane fiber particulates reinforced epoxy composites. Among all the sugarcane short fiber composites, 1wt% of sugarcane short fiber composites exhibited the highest tensile and flexural properties compared to 2.5wt%, 5wt% and 7.5wt%. Sugarcane fiber particulates reinforced composites were shown to have better performance when the wt% of the fiber increase while compared to short fiber reinforced composites.

CHAPTER 5
CONCLUSION

5.1 General conclusion

Due to the escalating problem arising from exploiting the natural resources, the utilization of natural fibers, such as sugarcane bagasse fibers (SCB) is favored in the manufacturing of composite. However, SCB alone as the sole filler is proven to be insufficient for enhancing the mechanical properties of composite. Hence, several fillers such as rice husk ash (RHA) and nanosilica are chosen to be the reinforcing medium to the primary medium, epoxy matrix.

Scientific significance of the research includes:

I) A novel bio-composite material with the combination of SCB fibers, Rice husk ash (RHA) and nanosilica particulates being incorporated into the epoxy resin. When SCB fibers are embedded into the epoxy in randomly distributed form, there are voids formed that are caused by the overlapping of fibers in the matrix. Hence, RHA in this case, will serve as the micro-filler, in order to fill the gaps in between the fibers. While nanosilica serve as the nano sized filler filling the nano-spaces in the composite system.

II) Agglomeration and uneven distribution of reinforcement in matrix often occur when too much fillers of one kind are embedded into the resin. Hence, when certain amounts of nanosilica and RHA are introduced into the resin, it is highly anticipated that the micro-voids in the epoxy resin can be reduced, while agglomeration can be minimized too.

5.2 Conclusion

SCB short fiber reinforced epoxy composites obtained higher flexural modulus compared to sugarcane fiber particulates reinforced epoxy composites. From the findings of the sugarcane short fiber composites, 1wt% of sugarcane short fiber composite exhibited the highest tensile and flexural properties compared to 2.5wt%, 5wt% and 7.5wt%. Sugarcane fiber particulates reinforced composites were shown to have better performance when the weightage of the fiber increase while compared to short fiber reinforced composites. When proper modification and manufacturing procedures are applied, composites reinforced with SCB showed improvement in the mechanical properties such as Young's modulus and flexural modulus.

However, when the percentage of SCB weightage increases, the mechanical properties of the composite were noticed to decrease significantly. Furthermore, SEM images which presents the fracture morphology of the specimens that are made of epoxy resin reinforced by SCB in fibrous form also shown the fiber pull outs of the composite that left with voids spaces in the composite surfaces.

Through this study, it was revealed that SCB alone is not eligible in enhancing the mechanical performance of the composite. An optimum fiber loading also plays an important role in achieving the desired mechanical properties. From the results of relatively poor mechanical properties, it is obviously proven that 5wt% and beyond of SCB reinforcement is not suitable to be implemented. The fibers are too compact to be compressed and dispersed within the epoxy matrix. Hence, the amount of matrix that being shared between the incorporated fibers for attachment became lesser and affected the mechanical performances of the composite.

Current study also involved the utilization of rice husk ash (RHA) and nanosilica as two other reinforcing fillers in the making of a hybrid composite. An important observation to be indicated was that the higher loading of RHA in the epoxy matrix

contributed to the Young's modulus of the composite. The Young's modulus of the composite NRHA750 is found to be highest among all the relative composite reinforced with RHA. The incorporation of RHA can fill in the spaces of micro-voids, thus improving the mechanical performance of the filler while the presence of nano silica particulates will contribute to the filling of nano-size voids. From the results, it can be concluded that the nano-silica particles have improved the mechanical performance especially tensile properties of the composite due to its large surface area and its smooth nonporous surface.

Theoretically, it was anticipated that when SCB, RHA and nanosilica are used in combination, the well dispersed RHA can fill up the micro-voids and nanosilica filling up the nano-sized spaces that have been formed in the epoxy matrix to grant the material with better resistance to inhibit the external stress; while SCB fibers aid in the transferring and bearing the undesired loads by failing (rupture) first rather than the matrix. Hence, the RHA, nanosilica and SCB fillers are added simultaneously into the epoxy matrix. Due to the entanglement of fibers in the resin, weak interfacial bonding between the filler and matrix in the composite and relatively rough surface finish of the composite affected the mechanical performance of the hybrid composite.

From the results, it is worth mentioning that all the sugarcane short fiber hybrid reinforced epoxy composites obtained higher flexural modulus compared to sugarcane fiber hybrid particulates reinforced epoxy composites. However, sugarcane fiber particulates reinforced composites were shown to have better performance overall when the weightage of the fiber increase when compared to short fiber reinforced composites.

5.3 Future Recommendations

There are few suggestions and improvements for future works have been identified throughout the course of study as follows:

- Other mechanical properties such as impact and hardness can be carry out for comprehensive investigation of composites.

- Current methodology involves only one kind of method, which is hand-layout method. Hence, more sophisticated methods such as injection molding and hot-press molding can be applied to accommodate more fibers at higher weightage.

- Out of auto-clave oven can be introduced in the curing process for the effort to produce composite materials of complex contours, shapes and sizes, and with little-to-no void content.

- Surface treatment, other than chemical treatment, such as plasma treatment and biological treatment of fiber can be studied in order to compare the interfacial bonding between fiber and matrix of different surface treatment.

- Study can be conducted to evaluate the influence of curing time and temperature on the composite mechanical performanc

APPENDIX

CENTRAL LABORATORY
Universiti Malaysia Pahang, Lebuhraya Tun Razak,
26300 Kuantan, Pahang Darul Makmur.
Tel : 09-5493351 Fax : 09-5493353
E-mail : ucl@ump.edu.my

CERTIFICATE OF ANALYSIS (COA)

To / Attn	Fong Ai Ling		
Address	Curtin University Sarawak, CDT 250, 98000 Miri, Sarawak, Malaysia		
Tel No	085-443939 / 0168767821	Fax No	
Sample Lab No	2017/271	No of sample	1

Sample marking : 2017/271 (1)
Sample description : Rice Husk Ash
Date of sample received : 13-06-2017
Date reported : 13-06-2017

RESULTS:

Elements

No	Parameter	Results	Unit	Test Method
1.	Silicon (Si)	24.08	%	Quantexpress (Full Analysis) by XRF S8 Tiger
2.	Potassium (K)	1.39	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Phosphorus (P)	0.42	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Calcium (Ca)	0.40	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Chlorine (Cl)	0.17	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	Magnesium (Mg)	0.07	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	Iron (Fe)	0.07	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Sulphur (S)	0.05	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Manganese (Mn)	0.05	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Zinc (Zn)	95	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Rubidium (Rb)	90	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Strontium (Sr)	42	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Copper (Cu)	30	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

Oxide

No	Parameter	Results	Unit	Test Method
1.	Silicon Dioxide (SiO2)	51.51	%	Quantexpress (Full Analysis) by XRF S8 Tiger
2.	Potassium Oxide (K2O)	1.68	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Phosphorus Pentoxide (P2O5)	0.96	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Calcium Oxide (CaO)	0.57	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Chlorine (Cl)	0.17	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	Sulphur Trioxide(SO3)	0.13	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	Magnesium Oxide (MgO)	0.12	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Iron Oxide (Fe2O3)	0.09	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Manganese Oxide (MnO)	0.06	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Zinc Oxide (ZnO)	0.01	%	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Rubidium Oxide (Rb2O)	98	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Strontium Oxide (SrO)	49	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Copper(II) Oxide (CuO)	38	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

The above analysis is based on the sample submitted by the customer.

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NORHASLINA ABU SAMAH
SCIENCE OFFICER

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